ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE ENGINEERING AND TECHNOLOGY

THERMAL AND MECHANICAL PERFORMANCE OF CEMENTITIOUS PCM COMPOSITES

Ph.D. THESIS

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Department of Civil Engineering

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

ÇİMENTOLU FDM KOMPOZİTLERİNİN ISIL VE MEKANİK PERFORMANSLARI

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OCAK 2020

Erman Yiğit TUNCEL, a Ph.D. student of ITU Graduate School of Science Engineering and Technology with student ID 501112015, successfully defended the dissertation entitled "THERMAL AND MECHANICAL PERFORMANCE OF CEMENTITIOUS PCM COMPOSITES", which he prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

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To my family,

FOREWORD

In this dissertation, I wanted to call attention to the importance of energy efficiency and sustainable building material design. I believe that in the near future, building materials which contribute to the utilization of solar energy will find more place in the sector and become the prevalent materials.

This research was conducted under the supervision of Assoc. Prof. Dr. Bekir Yılmaz PEKMEZCİ. I would like to convey my sincere appreciation to him for his helpful guidance and endless encouragement throughout this dissertation.

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December 2019 Erman Yiğit TUNCEL

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ABBREVIATIONS

ANOVA : Analysis of variance
Approx. : Approximately
AR : Alkali- resistant
C : Cement, samples in which only cement used as binder material
DSC : Differential Scanning Calorimetry
EG : Expanded graphite
EIA : Energy Information Administration
FA : Fly ash, FA ash containing samples
GGBS : Ground granulated blast furnace slag
GHG : Global greenhouse gas
GRC : Glass fiber reinforced cementitious composite
HVAC : Heating, ventilating and air conditioning
LAC : Lightweight aggregate concrete
LWA : Lightweight aggregate
LWTA : Lightweight thermal aggregate
OA : Orthogonal array
PCM : Phase change material
PP : Permeable porosity
S : Slag, slag containing samples
S/B : Sand/total binder
S/N : Signal-to-noise
SCC : Self-compacting concrete
SCM : Supplementary cementitious materials
SEM : Scanning electron microscopy
TES : Thermal energy storage
TESCM : Thermal energy storage cement mortar
TOU : Time-of-use
UN : United Nations
WA : Water absorption capacity by weight
WR : Water reducer admixture

SYMBOLS

C_p	**:** **S**pecific heat capacity
$C_{p,s}$	**:** **S**pecific heat capacity (Solid)
$C_{p,l}$	**:** **S**pecific heat capacity (Liquid)
$C_p(T)$	**:** Temperature dependent specific heat capacity
D	**:** Distance between loading points
h	**:** Thickness
m	**:** Mass
M_1	**:** Mass of the saturated and surface-dried aggregate in the air
M_2	**:** Mass of the pycnometer containing the sample of saturated aggregate
M_3	**:** Mass of the pycnometer filled with water only
M_4	**:** Mass of the oven-dried test portion in air
Q	**:** Heat
H	**:** The amount of heat
P_{max}	**:** Crushing load
R	**:** Thermal resistance
R^2	**:** Coefficient of determination
S	**:** Standard deviation
T_1	**:** Initial temperature
T_2	**:** Final temperature
T_{endset}	**:** Ending temperature of phase transition
T_{onset}	**:** Starting temperature of phase transition
T_{peak}	**:** Peak temperature of phase transition
T_m	**:** Melting temperature
U	**:** Thermal transmittance
W_f	**:** Specific fracture energy
W_{PCM}	**:** Weight percentage of PCM
$\bar{y}$	**:** Mean response
ΔH	**:** Enthalpy change
ΔH_{endoth}	**:** Phase change enthalpie for melting
ΔH_{exoth}	**:** Phase change enthalpie for solidification

ΔH_m : Enthalpy of transition

ΔH_{PCM} : Measured latent heat of PCM

ΔH_T : Theoretical latent heat

ΔT : Change in temperature

λ : Thermal conductivity

ρ_{rd} : Oven-dried particle density

ρ_{ssd} : Surface-dried particle density

σ_{crush} : Crusshing strength

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THERMAL AND MECHANICAL PERFORMANCE OF CEMENTITIOUS PCM COMPOSITES

SUMMARY

Technological advancements and innovations lead to continuous developments in building materials. Recent developments enabled materials to gain different features in addition to their main functions. In this way, it has become possible to respond to different needs with a single material. One of these technologies is the thermal energy storage (TES) technique. TES, which makes significant contributions to sustainable development, can be explained simply as the technology that stores thermal energy by heating or cooling a storage medium so that the stored energy can be used later for heating and cooling applications and power generation.

Energy is a central figure to nearly every major challenge and opportunity the world faces today. For many years, the greatest portion of the world energy demand has been provided from fossil-based fuels such as coal, oil or gas. Burning carbon fuels generates a considerable increase in the level of greenhouse gases which is the leading cause of global warming. In addition to all the negatives of using carbon fuels, the world is running out of its fossil fuel supplies. It is also a known fact that the energy consumption of the world has been increasing dramatically during the last century resulting a depletion in energy reserves and an increase in environmental degradation. Moreover, the rise in demand is expected to progress further in the near future. The search for alternative clean and renewable energy has now become the primary concern in all over the world, especially for the countries which import the demanded energy. Renewable energies like solar, geothermal, etc. are the most viable solution to the problem. The full implementation of such energy sources requires storage capabilities to be developed. For the case of the sun, solar energy can be directly utilized for heating or cooling purposes, such as heating for winter times and for domestic hot water needs or cooling in summer times through phase change materials.

This dissertation is planned as a cumulative work consisting of three published scientific articles and is framed within thermal and mechanical performance of the cementitious composites containing phase change materials (PCMs). A PCM is a latent heat storage material that undergoes a phase change with endothermic and exothermic reactions. In our studies, the goal is to develop building materials that support to the efficient use of energy with the use of PCM. In the first two publications (Chapter 2 and Chapter 3), lightweight aggregate production by pelletization method and integration of PCM into the method were examined. In the second article, concrete production using the manufactured aggregates and effects of the aggregate on the mechanical and physical properties of concrete were discussed. In the last article (Chapter 4), the thermal and mechanical performance of glass fiber reinforced cementitious composites (GRCs) with their enhanced mechanical strength and energy absorption capacity, was studied in the presence of microencapsulated PCM over different service temperatures. Finally, the dissertation was rounded out by the conclusions drawn from the studies and recommendations for future works.

Main focus of the first publication is the artificial aggregate production by pelletization which is the method that has been used throughout the study to integrate PCM to the building materials through the aggregates. The study has a pivotal environmental significance because it enables recycling of by-products such as FA and GGBS. The technique also contributes to the efficient use of energy in terms of suggesting a practical production process for manufacturing aggregates capable of TES. In the first stage of the study, the dynamics of the pelletization method was investigated. The effects of factors such as binding material content, amount of sand used, curing duration was revealed by using experimental design and optimization processes. The study showed that each material included in the mixture changes the amount of water needed for pellet formation which affects the engineering properties of the product. Modifications in the pore structure of the produced aggregates arising from different water amount in the mixtures, directly affected both the physical properties of the material such as density and permeability, and strength properties such as crushing strength. Use of sand in mixture facilitated the agglomeration. Binder type was found to be more effective on physical properties than S/B ratio according to Taguchi analysis. When GGBS was used as binder, lighter aggregates were obtained compared to C and FA binder ones. ANOVA results showed that S/B ratio has a significant effect on porosity of aggregates. The effect of binder type, S/B ratio and curing duration on crushing strength of aggregates were studied and the most significant factor was found to be the curing time. Since FA and GGBS are pozzolanic materials, aggregate strength showed significant changes with extended curing period depending on the type of binder used. It was obtained that the use of GGBS allows the production of lightweight aggregates with sufficient early strength. By incorporating a microencapsulated PCM into the production process, lighter and thermally enhanced aggregates were produced in the second stage of the research. Sand was also used in design of PCM aggregates because it helps to pellet formation. Although it was below the expected level, PCM addition improved the thermal capacity of aggregates. However, a significant decrease in strength with the increase of PCM amount was observed.

The second study showed the physical, mechanical and thermal properties of aggregates containing cement, FA and GGBS in a more detailed manner and gave the opportunity to evaluate the aggregate performances comparatively depending on the materials included. It was observed that when the PCM amount of the mixture is increased, the pelletization process takes longer and the mixture in the disk tends to sludge without any pellet formation. PCM use increased the water/binder ratio of the mixtures and this ratio increased in a linear manner by the increase of the PCM ratio, regardless from binder type. The increase of PCM volume in the mixture conduced the PCM particles to approach each other in the structure, causing the matrix phase to decrease in volume gradually. The PCM incorporation decreased the particle density gradually down to 0.99 g/cm^3. This means that it is possible to produce quite light aggregates using PCM. PCM additon affacted the water absorption capacity and porosity of the aggregates at different levels depending on the content of the mixtures. The study showed that the increase of PCM ratio of the aggregates generates a serious decrease in the crushing strength of the aggregates. This decline can be restrained by limiting the PCM ratio of the aggregates. Mechanical properties of aggregates were degraded regardless from binder type in case of PCM addition and the effect of PCM ratio on crushing strength of pellets was found to be more significant than the effect of binder type. A high correlation between the crushing strength and aggregate density has also been obtained. It can be concluded that, in general, as the density of the aggregates decreases based upon the increase of PCM ratio of the mixtures, the

crushing strength of the aggregates is also decreases. When the thermal performance of the aggregates was evaluated, it was seen that although PCM incorporation increased the latent heat capacity of the LWTAs and the increase was systematic, the observed enthalpies remained below the theoretical values. The limited PCM ratios of the aggregates and problems related to the test methodology can be accounted for this performance loss. As future work, LWAs with high porosity may be produced by pelletization method and after a desired strength development, a better thermal performance can be obtained by PCM impregnation method. In this way, sintering or hydrothermal treatment can be applied to aggregates to provide them extra strength prior to the PCM impregnation process. Concretes including produced LWTAs are another research topic discussed in the second article. Based on the experimental results, it has been seen that although the PCM causes a significant decrease in compressive strength of the concrete, it is still possible to produce a structural concrete with sufficient strength for many structural applications.

In the third study, in order to achieve a more advanced thermal performance, microencapsulated PCM was incorporated into GRC panels which offer a suitable layer covering exterior of the buildings for utilization of solar energy. The main idea of integrating PCM into the building materials is to be able to respond to a part of the building's energy needs by providing the energy storage feature to the existing elements of the building in addition to the building's own main functions. In cases where energy storage is not possible, integration of PCMs can also contribute to the insulation. By direct incorporation of the microencapsulated PCM into GRCs, a remarkable improvement was achieved in the thermal performance of composites. The study showed that the PCM in the cementitious composite affects the indoor temperature development successfully by blocking the excessive heat coming from the sun. Advanced heat capacity of PCM composite together with its relatively lower thermal conductivity improved the insulation properties of the material. It also changed the temperature acceleration and enabled shifting the time of the peak temperature to another time zone which offers a lower tariff. The PCM composites also maintained their performance even at temperatures above their phase transition temperature zone. In conclusion, fiber reinforced PCM composites increase the thermal efficiency of a building and reduce the cooling expenses by removing the electrical loads of the cooling systems especially during the summer. In addition to the improvements in thermal performance, the study focused on the losses in the mechanical performance of the material and questioned the change in mechanical performance after the phase transformation of the PCM within the GRC. It was observed that the decrease in uniaxial compressive strength of composites is higher than the decrease in flexural strength.

In conclusion, cementitious PCM composites is a promising material for sustainable buildings. These composites can reduce the energy consumption of a building and increase the thermal comfort. Considering the fluctuations at different service temperatures, the performance should be adjusted in a way that it does not fall below the design strength value for an optimum use of the material.

ÇİMENTOLU FDM KOMPOZİTLERİNİN ISIL VE MEKANİK PERFORMANSLARI

ÖZET

Teknolojik yeniliklere parelel olarak yapı malzemeleri de gün geçtikçe gelişmektedir. İlerleyen teknoloji, malzemelerin kendi ana fonksiyonlarının yanında farklı özellikler kazanmalarına da ön ayak olmaktadır. Bu sayede tek bir malzeme ile farklı amaçlara cevap vermek mümkün hale gelmiştir. Bu teknolojilerden bir tanesi de ısıl enerji depolamadır (IED). Sürdürülebilir kalkınma yolunda önemli katıkılar sağlayan IED, basitçe, depolanan enerjinin daha sonra ısıtma ve soğutma uygulamaları veya güç üretimi için kullanılabilmesi amacıyla, bir depolama ortamını ısıtmak veya soğutmak suretiyle ısıl enerjiyi depolayan teknoloji olarak açıklanabilir.

Enerji, dünyanın yüzleştiği her türlü zorluk ve fırsatın merkezinde olup üretiminden saklanmasına, erişilmesinden kullanılmasına, bütün aşamaları ile geçmişten günümüze ilgi odağı olmuştur ve gelecekte de olmaya devam edecektir. Günümüzde ihtiyaç duyulan enerjinin büyük bir kısmının kömür, petrol benzin gibi fosil tabanlı yakıtlardan sağlanması hem çevreye verilen zarar bakımından hem de bu kaynakların sonlu olması bakımından sürdürülebilirlikten uzak bir tablo oluşturmaktadır. Öte yandan, dünyadaki enerji talebinin geçtiğimiz yüzyıl boyunca belirgin bir şekilde artması ve bu artış eğiliminin yakın gelecekte de yükselerek devam etmesinin beklenmesi, temiz ve yenilenebilir enerji için alternatif strateji arayışını dünya gündemine oturtmuştur. Güneş ve yerısıl enerjiler gibi yenilenebilir enerjiler bu duruma en uygun çözüm yolunu oluşturmaktadır. Güneş; kış aylarında ısınma, yaz aylarında soğutma ve tüm mevsimlerde sıcak su gibi tüm termal enerji ihtiyaçları için iyi bilinen bir kaynaktır. Yenilenebilir enerjilerin tam olarak uygulanması için ısıl enerjinin depolanmasının şart olduğu da unutulmamalıdır.

İnşaat sektörünün dünya enerji tüketimindeki payı çok ciddi seviyelerde olup bu payın büyük bir kısmı ısıtma, soğutma, havalandırma (HVAC) gibi iklimlendirme işlemlerine aittir. Kentsel yenileme çabalarının hali hazırda devam ettiği kalkınmakta olan ülkeler için bina enerji verimliliğinin ön planda tutulması ve özellikle işletme giderleri başta olmak üzere enerji temelli harcamaların en aza indirilmesi oldukça büyük önem arz etmektedir. Bu nedenle, hem yeni inşa edilmesi planlanan hem de yenilenmesi söz konusu olan binalarda enerji verimliliğini arttıracak önlemlerin alınması ile bina temelli enerji tüketiminin sınırlandırılması, sürdürülebilir bir kalkınma stratejisi sağlamak açısından bina yapım yönetmelikleri ve hükümet kalkınma planlarında ön saflarda yer almaktadır.

Özellikle sıcak yaz aylarında insanların ısıl konforuna bir tehdit olarak ortaya çıkan aşırı güneş enerjisi, pasif bina konsepti çerçevesinde enerji israfı sorununa rasyonel bir çözüme dönüştürülebilir. Pasif binalarda, binanın duvar, döşeme veya pencere gibi unsurları, kış aylarında güneş enerjisini ısı şeklinde toplayacak, depolayacak ve dağıtacak; yaz aylarında ise yapıyı güneşten gelen fazla ısıdan koruyacak biçimde tasarlanmıştır. Pasif yöntemlerle, geleneksel malzemelerden daha üstün ısıl özelliklere sahip yapı malzemelerinden yararlanılarak daha az dış kaynaklı enerjiye ihtiyaç duyan

ve dolayısı ile daha konforlu bir yaşam ortamı sunan binalar inşa edilebilir. Yalıtılmış binalar, doğal havalandırma, trombe duvarı, uygun yapısal konumlandırma, bitki örtüsü yardımıyla gölgelendirme vb. yöntemler düşük enerjili ve yüksek konforlu binalara ulaşmada kullanılabilecek pasif yöntemlere örnek verilebilir. Binalarda güneş enerjisinin kullanılması, hem konut sakinlerinin enerji harcamalarını azaltacak hem de iç mekân termal konforlarını arttırmalarına yardımcı olacaktır.

Malzemelerde ısı enerjisinin depolanması, hissedilir ısı, gizli ısı ve kimyasal reaksiyonlar yoluyla sağlanabilir. Antik çağlardan beri, yapı malzemelerinin hissedilebilir ısı kapasitelerinin kullanımı, binaların ısıl konforlarını sağlamada süregelen bir yöntemdir. Ancak bu yöntemle kısıtlı bir enerjinin depolanması söz konusu olduğundan uygun bir ısıl konfor için oldukça geniş yapı kesitleri gerekmektedir. Modern yapılarda yüksek dayanımlı malzemelerin kullanılmaya başlanması ile daha ince kesitlere sahip duvarlar mümkün hale gelmiş ancak bu gelişme yapının ısıl kütlesinin azalmasına neden olmuştur. Bu da enerji ihtiyacı bir hayli fazla olan iklimlendirme sistemlerinin kullanımını kaçınılmaz hale getirmiştir. Yeni yüzyıldaki gelişmeler ile birlikte, yüksek gizli ısı kapasitesine sahip, faz değiştiren malzemeler (FDM) olarak adlandırılan maddelerin yapı sistemlerinde kullanımı ile daha ince kesitli ancak ısıl kütlesi yüksek yapıların inşası mümkün hale gelmiştir. FDM'ler endotermik ve egzotermik tepkimeler sonucunda faz değişimine uğrayan gizli ısı depolama malzemeleridir. Başka bir deyişle, faz değişim sıcaklıkları civarında bir ısı değişimine maruz kaldıklarında, ısı artışının söz konusu olduğu durunmlarda, bu malzemeler çevreden aldıkları ısı enerjisini faz değiştirmede kullanmak suretiyle katıdan sıvıya veya sıvıdan gaza, depoladıkları enerjiyi dışarıya verdiklerinde ise gazdan sıvıya veya sıvıdan katı hale geçerler. FDM aracılığıyla sadece hissedilir enerji depolanmaz, aynı zamanda gizli ısı depolanması söz konusudur. Bundan dolayı, FDM'nin gizli ısı kapasitesinin kullanımı ile su, taş duvar gibi hissedilir ısı depolama malzemelerine oranla birim hacim başına çok daha fazla ve izotermal özellikte ısı depolamak veya geri kazanmak mümkündür.

Elde edilmesinden kullanılmasına FDM'ler oldukça geniş bir araştırma alanı oluşturmaktadır. PCM'lerin yapı sektöründe kullanımı ise enerji verimliliği ve temiz enerjiye verilen önemin gün geçtikçe artması ile son yılların gözde araştırma konularından biri haline gelmiştir. Temiz ve verimli enerji kullanımı ile sürdürülebilir kalkınmaya verdikleri destek ile kullanıcılara ve ülkeye uzun vadede sağlayacakları ekonomik katkılar FDM'lerin yapı malzemelerine entegrasyonunu oldukça cazip kılmaktadır. FDM'nin yapıda kullanımının görece yeni bir araştırma alanı olmasının yanında bu alanın oldukça geniş sınırlara sahip olması, konu ile ilgili araştırma ihtiyacını arttırmaktadır. FDM olarak kullanılabilecek malzemelerin çeşitliliği, yeni malzeme keşfetme çabaları, mevcut ve yeni bulunan FDM'lerin yapı malzemeleri ile uyumu ve entegrasyonu, entegrasyon sonrası son üründe meydana gelen performans değişimleri, ekonomik kaygılar gibi konular düşünüldüğünde FDM'lerin yapıda kullanımı konusunun çok boyutluluğu konusunda bir fikir sahibi olunabilir. Genel olarak FDM'lerin ısıl depolama konusundaki başarıları literatürde mevcut birçok çalışma ile kanıtlanmıştır. Gerek FDM çeşitliliği gerekse FDM'nin entegre edilmesi planlanan yapı malzemelerinin ve yapı sistemlerinin çok çeşitli olması farklı FDM'lerin farklı sistemlerde kullanılabilirliğinin değerlendirilmesi gerekliliğini doğurmuştur. Bu bağlamda bu tezde ele alınan çalışmaların ana çerçevesini FDM içeren çimentolu kompozitler oluşturmaktadır. Tezin ana amacının en genel anlamda, enerji verimliliğine katkı sağlayan, yapısal anlamda yeterli, çimentolu kompozit malzemeler geliştirilmesi olduğu söylenebilir.

Literatürdeki çalışmalar incelendiğinde FDM'nin yapı sektöründe agregalardan sıvalara, harç malzemelerinden betona kadar birçok yapı malzemesi bünyesinde kullanıldığı görülmüştür. FDM'nin agrega bünyesine entegrasyonu ile harç, beton gibi yüksek oranda agrega içeren yapı malzemelerinin enerjinin verimli kullanımına destek verecek biçimde tasarlanmasına yönelik ilk adım atılmış olur. Betonların karışım tasarımları, kullanılacakları yer ve amaca göre çok çeşitli varyasyonlar gösterebilir. Piyasada her türlü beton dizaynı dahilinde kullanılmaya hazır standart bir FDM-agreganın bulunması kullanım kolaylığı bakımında pratik olarak sektöre yarar sağlayabilir. Literatür değerlendirildiğinde FDM'nin agrega içerisine genellikle emdirilme yöntemi ile entegre edildiği gözlemlenmiştir. Bu yöntemle oldukça başarılı ürünler geliştirilmiş olsa da eridiği zaman akışkan bir hal alan FDM'nin bulunduğu agrega boşluklarını terk etmesi akabinde yaşanabilecek FDM kusması, agreganın FDM içeriğinde kayıplar meydana gelmesi, mekanik ve geçirimlilik özelliklerinde değişiklik gibi problemleri akla getirmektedir. Bu bağlamda literatürdeki yöntemlere alternatif bir yöntem önermek amacıyla, çalışmada ilk olarak mikroenkapsüle FDM'nin peletleme yöntemi ile yapay agrega üretimine dahil edilmesi incelenmiş, uçucu kül, öğütülmüş yüksek fırın cürufu gibi endüstriyel yan ürünler kullanılarak yapay hafif agregalar üretilmiştir. FDM içeren hafif agrega üretimi ile enerji verimliliğine ek olarak hafif yapı malzemesi kullanımının bereberinde getireceği avantajlar ve uçucu kül, cüruf gibi Türkiye'de oldukça fazla açığa çıkan endüstriyel yan ürünlerin değerlendirilmesi amaçlanmıştır.

Üç farklı bilimsel yayının bir araya getirilmesi ile oluşturulan çalışmanın ilk ayağında soğuk bağlanmış hafif agregaların tasarımı, optimizasyonu ve performansı üzerinde durulmuştur. Agrega bileşiminin deneysel tasarımında Taguchi yöntemi kullanılmıştır. Agregaların mühendislik özelliklerini etkileyen faktörler ve katkı oranları, varyans analizi (ANOVA) kullanılarak incelenmiştir. Faktörlerin optimal değerleri belirlenmiş ve araştırmanın devamında ısıl performansı iyileştirmek adına ticari bir mikroenkapsüle FDM kullanılarak cüruf içeren agrega karışım tasarımı geliştirilmiştir. Farklı FDM oranlarının üretilen peletlerin mühendislik özelliklerine etkisi deneysel olarak incelenmiştir. Özellikle agregaların mekanik ve fiziksel özelliklerinde meydana gelen negatif etkilerin boyutu üzerinde durulmuştur. İkinci bilimsel yayın birincinin devamı niteliğinde olup bu çalışmada araştırmanın sınırları genişletilerek farklı FDM oranları ile tasarlanmış çimento, uçucu kül ve cüruf içeren yapay agrega grupları üretilmiş ve fiziksel, mekanik ve ısıl özellikleri deneysel olarak araştırılmıştır. Akabinde mühendislik özellikleri belirlenen bu agregalar kullanılarak muadil betonlar üretilmiş, üretilen agregaların beton özelliklerine etkileri incelenmiştir.

Çalışma sonunda karışıma dahil edilen her farklı malzemenin pelet oluşumu için gerekli olan su ihtiyacına etki etmek suretiyle pelet oluşumu ve ürünün mühendislik özelliklerini doğrudan etkilediği görülmüştür. Üretimde farklı oranlarda su kullanılması agregaların içyapısını etkileyerek hem fiziksel hem de mekanik özelliklerinde değişime sebep olmuştur. Çalışmanın bir diğer pratik sonucu, kumun pelet oluşumunu kolaylaştırması ve peletleme sürecini hızlandırmasıdır. Öte yandan, karışımdaki kum miktarı belirli bir eşiği aştığında, mevcut bağlayıcı miktarı tüm içeriği bağlamak için yetersiz hale gelmekte ve bu da peletleme diskinin tabanında bağlanmamış malzemelerin birikmesine neden olmaktadır. Agrega içeriğinde kum kullanımı ile ilgili ulaşılan bir başka bulgu ise kumun, üretilen agregaların yoğunluğunda bir miktar artışa neden olduğudur. Bununla birlikte, kumun, agrega dayanımında önemli bir artış oluşturmadığı da belirlenmiştir. Kum ile ilgili bulgular birlikte değerlendirildiğinde, çalışmanın ilerleyen safhalarındaki tasarımlarda

peletlemeyi kolaylaştırmak adına kum kullanımını minimum seviyede tutmanın optimum çözüm olacağı sonucuna varılmıştır. Taguchi ve varyans analizlerine dayanarak, bağlayıcı tipinin agrega yoğunluğu, su emme ve geçirimli porozite gibi fiziksel özellikler üzerinde en etkili parametre olduğu belirlenmiştir. Dayanım özellikleri söz konusu olduğunda ise, kür süresi en etkili faktör olarak bulunmuştur. Bu analizler sonucunda, bağlayıcı tipi ve kür süresi faktörlerinin etkileşiminin dayanım üzerinde oldukça etkili olduğu da görülmüştür. Çalışma aynı zamanda, çimento, uçucu kül, cüruf gibi farklı bağlayıcı türleri kullanılarak üretilen agregaların birbirleri ile kıyaslanmasına olanak vermiş, cüruf kullanılarak üretilen agregaların uçucu kül kullanılarak üretilen agregalara oranla daha hafif olduğu belirlenmiştir. Agregaların dayanım özellikleri göz önüne alındığında, cüruf agregalarının erken dayanımı, uçucu kül agregalarından daha yüksek olmasına rağmen, kür süresinin uzamasıyla birlikte, uçucu kül agregalarının nihai dayanım açısından daha iyi performans gösterdiği sonucuna varılmıştır.

Agrega tasarımlarına mikroenkapsüle FDM'nin eklenmesi ile genel olarak agregaların hafiflediği ve dayanım kaybettikleri, buna karşın ısı depolama kapasitelerinin arttığı söylenebilir. Karışımlara FDM eklenmesi peletleme işleminin süresinin uzamasına sebep olmuş, karışımların pelet oluşturma eğilimlerinde azalma gözlemlenmiştir. Uzun süren peletleme süreci nedeniyle agrega üretiminde kullanılan su miktarları da artış göstermiş, yüksek oranlarda FDM kullanımı söz konusu olduğunda dayanımda önemli mertebede düşüşler gözlemlenmiştir. Ayrıca, artan FDM ve su miktarı disk içersindeki karışımın çamur halini almasına sebebiyet vermiş bu durum agrega içerisinde yüksek miktarlarda FDM kullanımını engelleyerek, agregaların ısı depolama özelliklerini sınırlamıştır. Çalışmada elde edilen sonuçlara göre, agrega su/bağlayıcı oranının, bağlayıcı türünden bağımsız olarak, artan FDM oranı ile birlikte doğrusal bir şekilde arttığı söylenebilir. Çalışma ayrıca, incelenen etmenlerin agregaların mikro yapısında oluşturduğu farklılıkları da ortaya koymuştur. Karışımdaki FDM miktarındaki artış, matris malzemesinin yapısı ve hacmini de değiştirmiştir. FDM kapsüllerinin hacmi arttıkça parçacıklar birbirlerine yaklaşmış, matris hacmi artan FDM oranına paralel olarak kademeli olarak azalmıştır. Yüksek FDM oranına sahip agregaların kırılma yüzeylerinin birbiri ile temas eden FDM mikrokapsüllerinden oluştuğu, matris fazının oldukça sınırlı olduğu gözlemlenmiştir.

FDM'nin agregaların fiziksel özellikleri üzerindeki etkisi, araştırmada incelenen bir diğer konudur ve FDM kullanımının agrega yoğunluğunu 0,99 g/cm^{3}'e kadar düşürdüğü görülmüştür. Bu, FDM kullanarak oldukça hafif agregalar üretmenin mümkün olduğu anlamına gelmektedir. FDM, su emme kapasitesi ve poroziteyi karışımların içeriğine bağlı olarak farklı seviyelerde etkilemiştir. Karışımlardaki malzeme çeşitliliğinden kaynaklanan su oranlarındaki farklılıklar da bu sonuca neden olmaktadır. Sonuç olarak agrega içeriklerinin farklı olmasının çalışmanın genel sonuçları üzerinde zincirleme etkiler yarattığı söylenebilir.

Çalışma, agrega içerisindeki FDM oranı arttıkça, agregaların ezilme dayanımının ciddi bir şekilde düştüğünü göstermiştir. Bu sonuç agrega içerisinde kullanılabilecek FDM oranını sınırlayan başka bir etmen olmuştur. FDM agregaların mekanik özellikleri üzerinde bağlayıcı tipinden bağımsız olarak bozulmalara yol açmıştır. FDM'nin ve bağlayıcı tipinin dayanım üzerine etkisinin ayrı ayrı ele alındığı çalışmada, FDM kullanımının dayanım üzerinde bağlayıcı tipi etkisine göre daha yüksek mertebede bir etki yarattığı gözlemlenmiştir. Elde edilen sonuçlara değerlendirildiğinde, FDM oranındaki artış ile dayanım değerlerindeki düşüş arasında doğrusal bir ilişki vardır. Ezilme dayanımı ile agrega yoğunluğu arasında yüksek bir korelasyon olduğu da tespit

edilmiştir. Genel olarak, agrega yoğunluğunun, karışımların FDM oranının artmasına bağlı olarak azalması ile birlikte, ezilme dayanımının da düştüğü sonucuna varılmıştır.

Üretilen agregalarının ısıl performansı bu deneysel çalışma kapsamında değerlendirildiğinde, düşük oranlarda FDM kullanımının agrega ısıl performansını uygulamada fark yaratacak düzeyde yükseltmediği sonucuna varılmıştır. Dayanım, üretilebilirlik, ekonomi gib farklı kaygılar ile agregaların FDM oranlarının sınırlanması ve malzeme test sürecinin yarattığı problemler ısıl performansın istenen düzeye çıkarılamamasına neden olmuştur. Sonuç olarak FDM kullanımı agregaların gizli ısı kapasitesini arttırmış ve bu artış sistematik olarak elde edilmiş olsa da, gözlemlenen entalpiler teorik değerlerin altında kalmıştır. Gelecekteki çalışmalarda tam ölçekli deneysel analizler ile ısıl performans daha iyi irdelenebilir. Alternatif bir yöntem olarak, yüksek gözenekliliğe sahip hafif agregalar peletleme yöntemiyle üretildikten sonra istenen dayanım gelişimini takiben, agregalara FDM emdirme yöntemi denenebilir.

Çalışma ayrıca peletleme yöntemiyle üretilen agregalar kullanılarak hafif betonların üretilebileceğini göstermiştir. Beton içerisinde kullanılan agreganın FDM içeriği arttıkça betonun hem eğilme hem de basınç dayanımının düştüğü gözlemlenmiştir. Deneysel sonuçlar değerlendirildiğinde, FDM her ne kadar betonun basınç dayanımını olumsuz yönde etkilese de, bu agregalar kullanılarak birçok yapısal uygulama için yeterli dayanımda, yapısal bir beton üretilebileceği görülmüştür.

Üçüncü bilimsel yayında, daha gelişmiş bir ısıl performans elde etmek amacıyla mikroenkapsüle FDM, binaların dış cephelerini kaplayarak güneş enerjisinin değerlendirilmesine uygun bir ortam hazırlayan lif takviyeli çimentolu kompozit panellerin (GRC) yapısına dahil edilmiştir. FDM'nin GRC bünyesinde kullanımıyla, kompozitlerin termal performansında kayda değer bir gelişme sağlanmıştır. Çalışma çimentolu kompozit içerisindeki FDM'nin güneşten gelen aşırı ısıyı engelleyerek iç ortam sıcaklık gelişimini başarıyla etkilediğini göstermiştir. Kompozitlerin gelişmiş entalpileri, düşük ısıl iletkenlik özelliği ile birlikte malzemenin yalıtım özelliklerini iyileştirmiştir. Ayrıca FDM'nin çalışma dahilinde üretilen ölçüm hücrelerinin sıcaklık artış ivmesini değiştirdiği ve hücreler içerisinde en yüksek sıcaklığın gözlemlendiği zamanını ileriye ötelediği gözlemlenmiştir. Sonuç olarak, lif takviyeli FDM kompozitlerinin yapıda kullanımı, yapının ısıl verimliliğini artıracak ve özellikle yaz aylarında soğutma sistemlerinin elektrik yüklerini azaltarak soğutma giderlerini düşürecektir. Isıl performanstaki gelişmelere ek olarak çalışma, malzemenin mekanik performansındaki kayıplar üzerine yoğunlaşmış ve GRC içerisindeki FDM'nin faz dönüşümünü takiben mekanik performanstaki değişim incelenmiştir. FDM kullanımı, kompozitlerin basınç ve eğilme dayanımlarında düşüşe neden olmuş, basınç dayanımının eğilme dayanımına göre daha yüksek oranda etkilendiği gözlemlenmiştir.

Sonuç olarak, çimentolu FDM kompozitleri sürdürülebilir binalar için umut verici bir malzemedir. Bu kompozitler binanın enerji tüketimini azaltır ve termal konforu arttırır. Ancak kullanım esnasında güvenli tarafta kalmak adına, yapının servis ömrü süresince maruz kalacağı farklı sıcaklık değerleri göz önüne alınmalı, malzeme performansı tasarım dayanım değerinin altına düşmeyecek şekilde ayarlanmalıdır. Gelecekteki araştırmalar için, bu araştırmanın kapsamına dahil edilmeyen, FDM içeren çimentolu kompozitlerin ekonomik uygulanabilirliği konusunu incelemek yerinde olacaktır. Gerçek ölçekli testleri içeren deneysel çalışmalar da konunun daha iyi anlaşılması açısından uygun olacaktır.

1. INTRODUCTION

1.1 Motivation and Background

Sustainable development, which has been defined as “development that meets the needs of the present without compromising the ability of future generations to meet their own needs” by the United Nations (UN), calls for concerted efforts towards building an inclusive, sustainable and resilient future for people and planet. In 2015, countries adopted the 2030 Agenda for Sustainable Development and its 17 Sustainable Development Goals (Figure 1.1) in the UN Sustainable Development Summit [1]. Energy, with many different aspects, is an essential element in this list.

Figure 1.1 : Sustainable development goals declared by UN [2].

Energy is central to nearly every major challenge and opportunity the world faces today. Figure 1.1 shows that one of the main topics in the Sustainable development goals declared by UN [2] is “affordable and clean energy” which aims to ensure universal access to affordable, reliable, sustainable and modern energy. For many years, the greatest portion of the world energy demand has been provided from fossil-based fuels such as coal, oil or gas, but burning carbon fuels causes a considerable increase in the level of greenhouse gases which promotes global warming leading climate change and harms people’s well-being and the environment [3]. In addition to

that, the world is running out of fossil fuels and the supply is not economically reliable [4], [5]. On the other hand, it is a known fact that the energy consumption of the world has been increasing dramatically during the last century, resulting a depletion in energy reserves and causing environment degradation. Moreover, this increasing trend is expected to progress further in the near future [6]. Search of the alternative strategies for clean and renewable energy has now become the primary concern all over the world, especially for the countries importing the demanded energy. Renewable energies like solar, geothermal, etc. are the most viable solution to the greenhouse gas problem. The sun can be used as the source of the many of the thermal energy needs such as heating for winter times, cooling for summer times, and domestic hot water for all seasons. It should be noted that the storage of the thermal energy is essential for the full implementation of renewable energies [7].

The construction sector has a significant share in the world's energy consumption and a large portion of this usage can be attributed to climatization operations such as heating, ventilating and air conditioning (HVAC) [8], [9]. For developing countries in which the urban renewal efforts are already in progress, it is essential to focus on energy efficiency and precautions must be taken in new constructions in order to reduce energy costs, particularly the operational ones. Therefore, limitation of the building based energy consumption by improving the energy efficiency of both new and refurbished buildings, remains at the forefront of the governmental plans and building codes in order to ensure a sustainable development strategy [10]. Design of energy efficient living spaces is also closely related to the goal 11 and 12 given in Figure 1.1, which are the constitution of sustainable cities and communities and the accomplishment of responsible consumption and production, respectively. By designing building materials that support energy efficiency and increasing the use of them in the building sector, sustainable cities can be created. When the innovative approaches used in building materials are combined with the philosophy of recycling waste materials, remarkable steps can be simultaneously taken for both responsible consumption/production and the urban transformation for sustainable cities.

Recycling of wastes is another issue that needs to be considered to ensure a resilient and sustainable city growth. Solid wastes, which is a particular concern of both urban and industrial life, is an ever-growing problem not only with its environmental aspect but also with its economical impacts since it constitutes a considerable burden on

goverments during the removal and management processes. Fly ash (FA) and ground granulated blast furnace slag (GGBS) are the industrial by-products which have a serious global production potential, especially in developing countries like Turkey. Although these products find themselves a place in cement and concrete sectors, the utilized material corresponds to only a small proportion of the total amount [11], [12]. These residuals can also be utilized in artificial aggregate manufacturing [13]. With the rigorous environmental restrictions imposed on the mining process in various parts of the world, the recovery of by-products has become an essential goal in the mining of natural aggregates. Utilization of these by-products also enables the conservation of natural aggregate resources and prevention of the damage caused by aggregate mining. Moreover, it is possible to produce lightweight aggregates (LWAs) by artificial manufacturing. LWAs bring some advantages to the construction sector such as smaller structural elements resulting in decreased dead loads and larger spans for use. Decreased element size also leads to reductions in both cement dosages and reinforcement. Transportation and handling expenses of lighter precast elements will be less. Elevated thermal insulation and improved fire resistance are the other benefits of artificial LWAs [14].

The research carried out in this dissertation is framed within thermal and mechanical performance of the cementitious composites containing phase change materials (PCMs). A phase change material is a latent heat storage material that undergoes a phase change with endothermic and exothermic reactions. In other words, in case of a heat exchange near its phase change temperature, it changes form from solid to liquid or liquid to gas with the increase of heat and from gas to liquid or liquid to solid giving its heat to the medium. In the research, with the use of PCM, it is aimed to develop building materials that support to the efficient use of energy. Thermal performance enhancement of building materials via PCMs is a worldwide active area of research. This research focuses particularly on artificial aggregates and glass fiber reinforced cementitious composites (GRCs), both containing PCM. In the manufacture process of the artificial aggregates, an eco-friendly approach has been adopted by utilizing industrial by-products such as FA and GGBS, thus contributing to the recycling of waste products. In the following sections, pelletization method which is used in the artificial aggregate production is summarized following the brief information about thermal energy storage (TES) by building materials and PCMs.

1.1.1 Passive building concept and TES in buildings

Excessive solar heating, which reveals as a threat to the thermal comfort of humans especially in hot summer months, can be utilized in the concept of the passive building. In passive buildings, the elements of the building like walls, floors or windows are designed in such a manner that they collect, store and distribute solar energy in the form of heat during the winter and reject solar heat in summer. Buildings designed with passive methods can have reduced energy consumption through utilization of building fabric which are thermally superior to the conventional fabric. Consequently, passive buildings offer a more comfortable built environment. Insulated buildings, natural ventilation, trombe wall, appropriate orientation of the building, vegetation causing shade etc. can be given as passive methods that can be used to obtain low-energy and high-comfort buildings [15], [16]. Use of sustainable solar energy in buildings will both reduce the energy expenditure of the residents and it helps them to increase the indoor thermal comfort.

The improvement of building materials continues day-by-day in parallel with the technological developments. Although the main function of a structural material is to carry the encountered load safely, advanced technology equips materials with new features and some additional abilities. One of these abilities is TES which is very contributive to abovementioned sustainable development goals. TES can be explained simply as the technology that stores thermal energy by heating or cooling a storage medium so that the stored energy can be used later for heating and cooling applications and power generation [17]. By utilization of building materials capable to store solar thermal energy, a great contribution to all three components of sustainable development-economic development, social development and environmental protection will be made. From the energy efficiency point of the view, these materials can provide a serious contribution to decrease the amount of energy used in residential and industrial life by virtue of their thermal properties.

The storage of thermal heat in materials can be provided by means of sensible heat, latent heat and chemical reactions. Heat storage as the sensible heat of building materials has always been a conventionally used method to utilize the thermal energy. The mathematical expression for sensible heat storage is given in equation 1.1,

$$Q = mC_p \Delta T \tag{1.1}$$

where Q is the heat stored (J), m is the mass (kg), C_p is the specific heat capacity ($Jkg^{-1}K^{-1}$) and ΔT is the change in temperature (K). Since for most materials C_p is relatively small (typically less than 2 $kJ\ kg^{-1}K^{-1}$) and ΔT is mostly fixed by the thermal comfort requirements, the mass of the building material must be large to store an appreciable amount of heat. This means that, very thick walls are required to procure satisfying amount of energy with sensible heat storage. Usually the amount of heat stored is expressed independent of mass as seen in equation 1.2,

$$\Delta H = \int_{T_1}^{T_2} C_p(T)dT \tag{1.2}$$

where ΔH is the enthalpy change in $kJ\ kg^{-1}$, and $C_p(T)$ is the temperature dependent specific heat capacity, so that an energy storage density can be estimated. The constant pressure heat capacity, C_p, is used here because the pressure remains relatively constant in almost all normal applications of thermal storage materials. Along with the developments of the new century, the use of latent heat capacity of materials in building sector has enabled the more modest structural cross-sections. Latent heat can be defined as the energy required to change the phase of a material [18] and the amount of heat that a latent heat storage material can store over a temperature range, T_1 to T_2 which includes melting, is given by equation 1.3,

$$\Delta H = \int_{T_1}^{T_m} C_{p,s}(T)dT + \Delta H_m + \int_{T_m}^{T_2} C_{p,l}(T)dT \tag{1.3}$$

where T_m is the melting temperature (K), $C_{p,s}$ and $C_{p,l}$ are the solid and liquid heat capacities respectively ($Jkg^{-1}K^{-1}$), and ΔH_m is the enthalpy of transition ($JkgK^{-1}$) [19], [20], [21]. Thermal energy storage through PCMs exploits latent heat along with sensible heat. Therefore, with the utilization of the latent heat of a PCM, much more heat storage per volume and isothermal heat storage or recovery may be possible in comparison with sensible storage materials like water, masonry etc. In Figure 1.2, change of the temperature with the stored heat in material is shown. As seen from the figure, temperature remains constant during phase transition [22], [23].

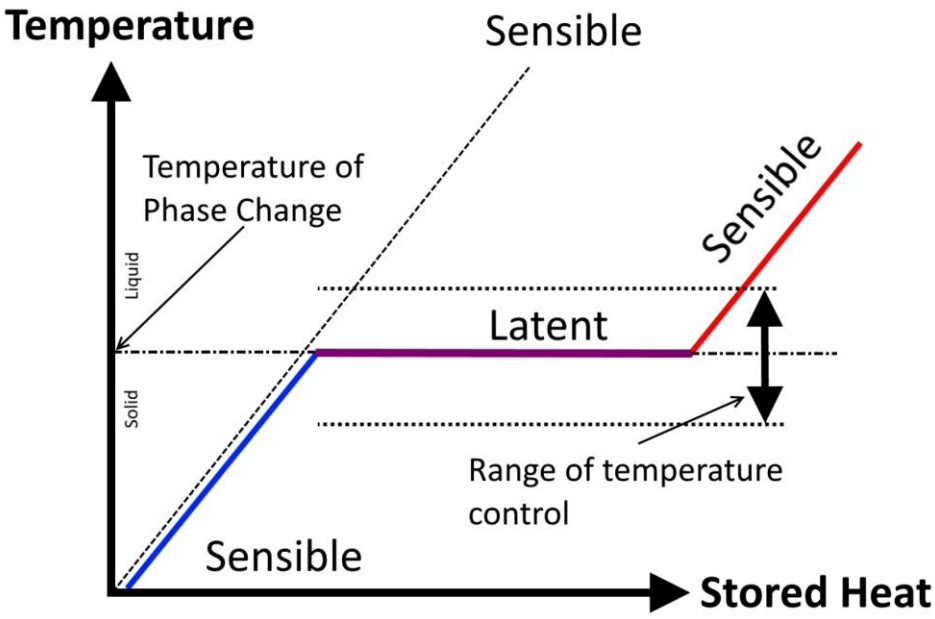


Figure 1.2 : Sensible heat vs. latent heat and temperature control during the phase change [23].

1.1.2 PCMs in buildings

Historically buildings were constructed considering both mechanical stability and indoor thermal comfort by keeping indoors warm in winter and cool in summer. The thermal comfort of the building was provided with walls having comparatively wider cross-sections. In a similar manner to the wider walls of the historical buildings, PCMs enable the use of the solar heat more efficiently and goal directed by means of their latent heat capacity. Working principle of a PCM in a building material can be explained by alternating phase transition cycles occurring within the material. In a given day, when the ambient temperature rises enough to reach the solid–liquid transition temperature, the PCM, incorporated in a construction element, changes from solid to liquid with endothermic behaviour (latent heat) thus limiting the flow of heat towards the interior of the building. Conversely, upon an environmental temperature decrease, the PCM that is now in the liquid state, may reach the liquid–solid transition temperature (melting temperature) again and shift to solid state, with energy release (exothermal process), thus delaying the cooling tendency inside the building [24].

Several materials can be used as PCMs. Any material with a melting point is a potential candidate, but the preferred materials have the phase transition temperature in the desired operating temperature range. In other words, the melting temperature should be consistent with the temperature required for the application in which the PCM will be used. Table 1.1 summarizes the preferred characteristics of PCMs.

Table 1.1 : Main desirable characteristics of PCMs [25].

Thermal properties	Physical properties	Chemical properties	Economic factors
Phase change temperature suitable to the desired operating range	High density	Chemical stability	Available in large quantities
High latent heat per unit mass	Low density variation during phase change	No chemical decomposition	Inexpensive
High specific heat	Little or no supercooling during freezing	Compatibility with container materials	
High thermal conductivity in both solid and liquid phases		Non-poisonous, non-inflammable and non-explosive	

PCMs can simply be classified into organics, inorganics and eutectic materials. These major groups together with their subgroups can be seen in Figure 1.3 [7]. In Table 1.2, some of the substances used in the literature is also presented [25]. Although the inorganic PCMs have higher volumetric latent heat storage capacity and thermal conductivity than the organic compounds, they are more corrosive to metallic materials (vessel containers) and subcooling may adversely influence their phase change properties. On the other hand, organic compounds are massive storage materials exhibiting reproducible melting and crystallization behaviour after a high number of thermal cycles. They are non-corrosive and chemically stable too. Flammability of some organic PCMs and having low thermal conductivity may be denoted as shortcomings of this group. Advantages and disadvantages of organic and inorganic PCM are summarized in Table 1.3 [7]. The last PCM group is eutectics which may be composed of organic-organic components or inorganic-organic components. A eutectic is a minimum melting composition of two or more components, each of which melts and freezes congruently. During the crystallization phase, a mixture of the components is formed, hence acting as a single component. [26].

Moreover, in order to improve some of the PCM properties, or to prevent corrosion, composite PCM, or also called hybrid, are being developed. Encapsulated PCM is one of the most common hybrid PCMs in practice. [27]. Within the available hybrid PCM, several final products can be found, called: shape-stabilized, slurries, microcapsules, etc. [28], [29].

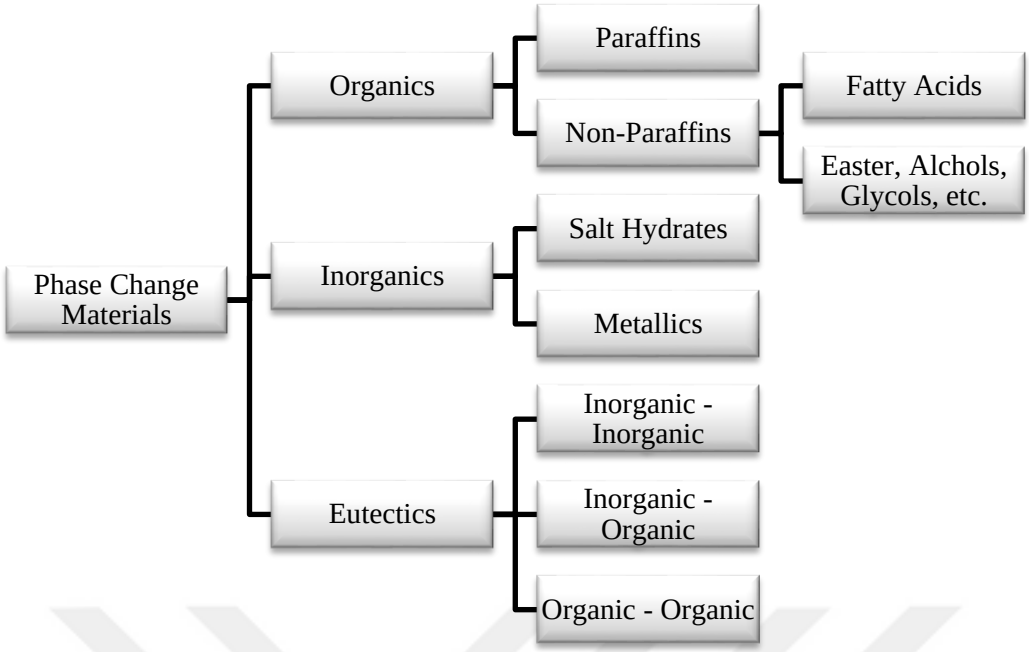


Figure 1.3 : Classification of phase change materials [7].

Table 1.2 : PCM examples used in the literature [25].

Organic substances	Inorganic substances	Fatty acids
Paraffin C_{13}	$Mn(NO_3)_2.6H_2O$	Capric-lauric acid (45-55%)
1- Dodecanol	$CaCl_2.6H_2O$	34% Mistiric acid + 66% Capric acid
Paraffin C_{18}	$LiNO_3.3H_2O$	Vinly stearate
1-Tetradecanol	$Na_2SO_4.10H_2O$	Capric acid
Paraffin C_{16-28}	$Na_2CO_3.10H_2O$	Lauric acid
Paraffin wax	$CaBr_2.6H_2O$	Myristic acid
	$Na_2HPO_4.10H_2O$	Palmitic acid
	$Zn(NO_3)_2.6H_2O$	Stearic acid
	$K_3PO_4.7H_2O$	
	$Zn(NO_3)_2.4H_2O$	
	$Na_2HPO_4.7H_2O$	
	$Na_2S_2O_3.5H_2O$	
	$Zn(NO_3)_2.2H_2O$	

Table 1.3 : Advantages and disadvantages of organic and inorganic PCM [7].

	Organic	Inorganic
Advantages	No corrosives Low or no subcooling Chemical and thermal stability	High phase change enthalpy High thermal conductivity
Disadvantages	Low phase change enthalpy Low thermal conductivity Flammability	Subcooling Corrosion Phase separation Phase segregation, lack of thermal stability

In the studies within this dissertation, a commercial, paraffin-based powder-type microencapsulated PCM (Micronal DS 5040 X) has been used. Considering the

potential workability problems which may rise from the use of a raw PCM along with cementitious materials, it has been deemed suitable to use a microencapsulated PCM within the work. Besides, paraffin based PCMs are prominent materials for the applications containing cement given their stability in alkaline environment and resistance in the high pH mediums associated with concrete [30].

1.1.3 Artificial aggregates and pelletization method

Aggregate is a collective term for the relatively inert mineral materials such as sand, gravel and crushed stone that are used with a binding medium such as water, Portland cement, lime, bitumen, etc., to form composite materials such as Portland cement concrete and bituminous concrete [31]. It is also used for base and subbase courses for both flexible and rigid pavements. Aggregates can be either natural or manufactured. Manufactured aggregate is often the byproduct of other manufacturing industries. Aggregates which contain byproducts (blast-furnace slag, cinders etc.) or manufactured materials (expanded clay or shale, processed diatomaceous earth, processed volcanic glasses, expanded slag etc.) are called *artificial aggregates* [32].

Process principles in artificial aggregate manufacturing can be specified as (i) mixing of raw materials, (ii) agglomeration, (iii) hardening or binding of the particles, (iv) further processing. Figure 1.4 shows these process steps in more detail [33].

Agglomeration is the term used for the entire field of consolidation of solid particles into larger shapes. Agglomeration of fines can be achieved with either agitation granulation or compacting. With compaction agglomeration, the density of the pellets will in general be higher than that of pellets produced with agitation granulation. Therefore, the granulation techniques, in which tumbling and capillary forces are employed, are relatively more suitable for lightweight aggregate manufacturing. In this technique, agglomeration is achieved by means of a disc (or pan), a drum, a cone or a mixer. Wetting is required to lump fine particles together with cohesion forces. Usually water is applied as a bonding agent but also other materials like waterglass, starch solution, emulsions of mineral oil in water or wastewater of paper mills are used as well. As the amount of liquid is increased in the mixture, the cohesion forces between the particles increase. There are three characteristic states of cohesion: (i) pendular state (ii) funicular state and (iii) capillary state, where the liquid amount in pores increases (Figure 1.5) from (i) to (iii). One critical factor for the pelletization process

is the dosage of the bonding agent; as too little or too much bonding agent means that insufficient cohesion forces can be developed or muddy balls instead of pellets might start forming [33].

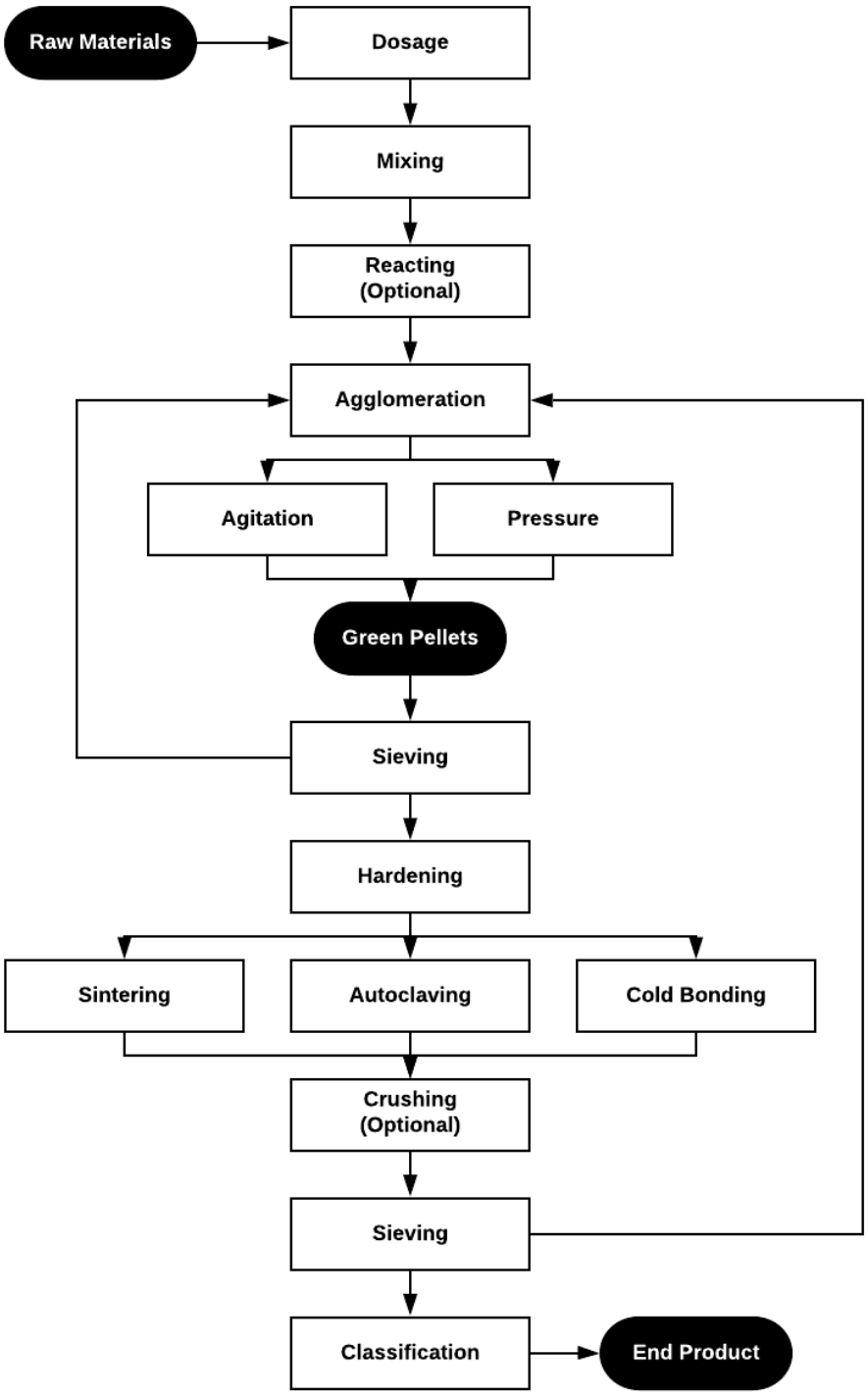


Figure 1.4 : Process steps in artificial aggregate processing [33].

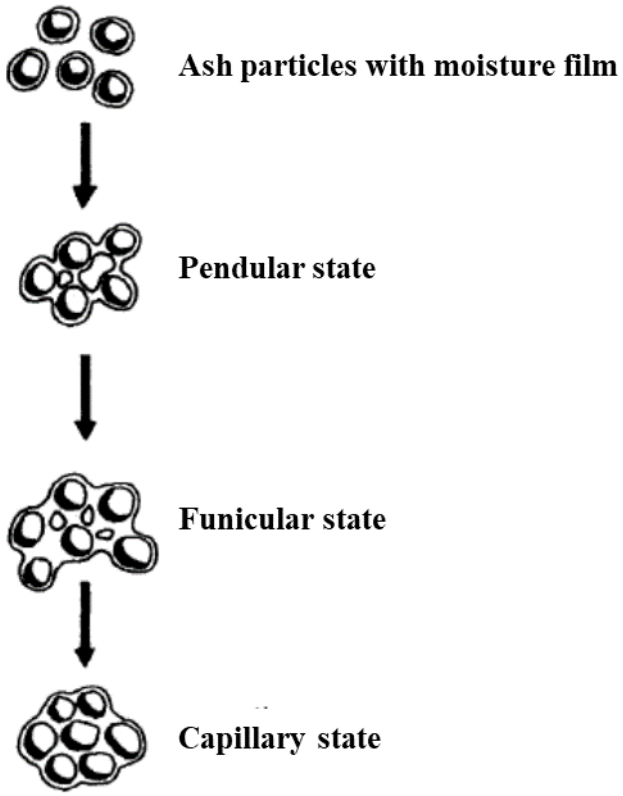
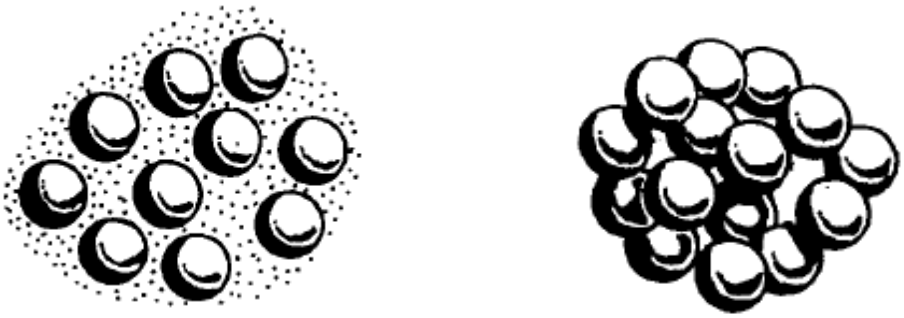


Figure 1.5 : Diagrammatic representation of pellet formation [33].

There are in general two types of bonding regarding hardening: bonding by a material bridge, and embedding in a matrix formed by reaction of a bonding material with part of the fly ash particle (Figure 1.6). Bonding by a material bridge can be achieved in sintering processes, which is based on fusing the particles together at the points of mutual contact, while there are quite a number of possible binders for matrix bonding. Bonding materials such as lime, cement and calciumsutphate/sulphite are the most common ones because of cost related and technical reasons [33]. Based on the temperature used, there are different hardening processes used in practice such as cold bonding (matrix bonding), hydrothermal treatments (matrix bonding under pressurized steam conditions) and sintering (material bridge bonding). In this dissertation, since microencapsulated PCMs are involved in the study, sintering method has not been preferred; instead, the strength gaining of the aggregates has been achieved via cold binding method. In this method, the ability of pozzolanic ingredient to react with calcium hydroxyde at ordinary temperatures is utilized to form a water-resistant bonding material. Lime or Portland cement are the source of calcium hydroxyde. Calcium sulphate, calcium sulphite and alkaline substances may have a positive effect on strength development. CSH-gel is formed and is the main bonding constituent.



(a) (b)

Figure 1.6 : Bonding types for agglomeration (a) Matrix bonding (b) Material bridge bonding [33].

1.2 Research Objectives and Questions

From procurement to application, PCMs have occupied a considerably large place in the scientific community. Utilization of PCMs in construction sector has become one of the most popular research topics of recent years in parallel with the increasing importance given to the clean and efficient energy use. During the last decade, subsidies given to clean and energy efficient buildings by governments has increased the demand for sustainable construction techniques. Efforts for integrating PCMs into buildings have gained a pace in parallel. The use of PCM in buildings is a relatively new field of research and has fairly wide boundaries and fundamental challenges that can be listed as: finding the right material from a large parameter space and the need for new materials; the compatibility and integration of PCMs with building materials; performance changes in the final product after integration and cost reduction for profitability.

In general, the success of PCMs in thermal storage has been proven by many studies in the literature. Both the diversity of PCM as well as the wide variety of building materials or building systems that the PCM can be integrated into, have generated a need to assess the availability of different PCMs in different systems. In this context, cementitious composites containing PCM constitutes the main framework of the studies discussed in this dissertation. The main aim of the dissertation in the most general sense can be declared as to develop structurally sufficient cementitious composite materials which contribute to energy efficiency. The research addresses two main topics in particular which are (i) artificial aggregates containing PCM and (ii) glass fiber reinforced cement composites containing PCM.

PCMs have been utilized in many building materials from aggregates to plasters, mortar materials and concrete. Since the building materials like mortar and concrete have a high aggregate content, integration of PCMs into the aggregate might be seen as the first step towards energy efficient construction designs. Concrete mix designs may vary depending on the place and purpose of use. The availability of a standard PCM-aggregate ready for use in any concrete design on the market can provide practical benefits to the sector in terms of ease of use. PCM is generally integrated into the aggregate by impregnation method as shown in previous studies. Although successful products have been developed with this method, PCM bleeding, which may occur as the liquid PCM leaves the aggregate pores right after the melting, suggests some problems such as reduction in the PCM content of the aggregate, alterations in mechanical and permeability properties. Thus, the first research question that sets ground for an alternative and original study was formed:

RQ 1.1: What alternative methods can be used to integrate PCM into aggregates? Is it possible to manufacture artificial aggregates containing PCM by the pelletization method?

The main motivation that lies behind this research question is the fact that the aggregate manufacturing by pelletization enables recycling of industrial by-products such as fly ash which are highly encountered in Turkey. In this context, the integration of PCM into artificial aggregates, which are produced using waste materials such as FA, GGBS, will yield multiple positive results. In the simplest sense, the production of artificial aggregates will create a positive environmental impact by preventing the rapid depletion of gradually decreasing natural aggregate reserves, and will make room for the building sector of many countries which faces with the harsh protection measures against aggregate mining. Recovering waste materials by the production of artificial aggregates will help governments in matters such as disposal of excess materials, storage costs and waste management. Moreover, the combined use of fly ash and PCM will allow the production of lightweight aggregate, which will provide benefits such as reduced building dead load, smaller structural elements, wider spans for use, and reductions in both reinforcement and cement dosage. The transportation costs of the lightened precast elements will also be lower. In addition, the use of lightweight aggregate will provide a better thermal insulation and fire resistance. The

use of PCM in aggregate will provide support for aforementioned clean energy issues and in this regard, the study will serve for sustainable development goals.

When the integration of PCM into artificial aggregate production through pelletization method is achieved, performance concerns about the production will arise. These concerns raise the second research question:

RQ 1.2: What are the possible design parameters that affect aggregate performance in artificial aggregate production? How does the use of PCM in mixtures affect the engineering properties of artificial aggregates?

The aim of this research question is to provide a better understanding of the dynamics of the pelletizing method and to provide an aggregate design that can be used as the basis for the formation of desired PCM aggregates. The effect of some prescribed factors on the engineering properties of the aggregates has also been investigated within the scope of this research question. To that end, the aggregate design has been optimized by using a Taguchi approach. After determining the most suitable aggregate mixture for the purpose, PCM supplement has been integrated into the study and mechanical, physical and thermal performance of cold bonded PCM aggregates has been investigated.

After determining the engineering properties of the PCM aggregates, the behaviour of these aggregates in concrete was questioned, which has generated the third research question:

RQ 1.3: Is it possible to design a structural concrete containing the aggregates produced in the study? How does the performance of the concrete change, based on the used aggregate in it? In other words, what are the effects of alternative aggregate properties on the concrete performance?

Considering the fact that artificial aggregates are designed to be used in concrete, it needs to be clarified whether the final product can lead to practical structural concrete. Our aim is to investigate the effects of artificial aggregates with different PCM contents on the physical and mechanical properties of concrete.

In the next stage of the research, cementitious PCM composites produced with the direct incorporation of microencapsulated PCM has been investigated in order to achieve a more advanced thermal performance. The main factor in examining this issue is that the cementitious composite panels, which form a layer covering the entire

surface of the structure, constitute a very attractive geometry that enables utilization of solar energy. When evaluated within the frame of passive building concept, cementitious PCM composite panels covering the building surface are very convenient and practical building components for TES. GRCs are very popular materials, especially in cladding applications because of their excellent mechanical properties, manageable consistency, and high fire and corrosion resistance. Although there is a common conclusion in literature that PCMs adversely affect the mechanical properties of building materials, the effects of PCM on fiber-reinforced cementitious composites is a topic that needs extensive research. Since PCM is incorporated into the composite to improve thermal storage, the first question that comes to mind is related to the thermal performance of composites:

RQ 2.1: Is it possible to make a meaningful contribution to the thermal performance of GRCs via microencapsulated PCM? What is the extend of this contribution and how does it change with the amount of PCM that is used in the composite?

The main idea here is to be able to respond to a part of the building's energy needs by providing the energy storage feature to the existing elements of the building in addition to the building's own main functions. In cases where this is not possible, additional insulation performance can be achieved with this application. With PCM containing materials of the building contributing to the indoor temperature control, the load of the active air conditioning systems will decrease.

After demonstrating the success of PCM on enhancing the thermal performance, the effect of PCM additive on the mechanical properties of the building material should also be questioned:

RQ 2.2: What is the effect of PCM on the mechanical performance of GRCs? How does it change with the amount of PCM that is used in the composite?

Although it is known that PCMs negatively affect the strength of building materials, it is essential for safety to consider this effect in detail for each designed building material. GRCs are materials recognized for their enhanced mechanical strength and energy absorption capacity. Especially, the variation of the flexural performance of these materials in the presence of PCM is one of the important properties that will affect their practical use.

Since the TES concept based on the latent heat is associated with phase transition of PCM, for each possible phase of the PCM contained in the material, a strength assessment of the material is required for safety. In other words, the mechanical performance of the PCM integrated building materials must be analyzed over different service temperatures that the temperature remains on either side of the phase change temperature value of the PCM. This requirement constitutes another research question:

RQ 2.3: Considering the varying service temperatures to which the material will be exposed, what are the strength fluctuations that may occur in GRCs? What are the upper and lower strength limits of the products examined in the study?

This question might be answered by testing two identical samples at varying temperatures that are above and below the melting temperature of the PCM. The transition of solid phase of PCM used in the material to the liquid phase must be considered when the mechanical performance of the final material is examined. For a safe design, the service temperature range of composites to be produced using PCM should be determined carefully and realistically, and the performance changes in composites should be fully revealed by carrying out experimental studies considering the most adverse conditions before use.

1.3 Structure of the Dissertation

The thesis is planned as a cumulative dissertation consisting of three published scientific articles. In the first two papers given in Chapter 2 and Chapter 3, lightweight aggregate production by pelletization method and integration of PCM into the method were examined. In the second article, besides the aggregate manufacture, concrete production using these manufactured aggregates and effects of aggregate properties on the mechanical and physical properties of concrete were discussed. In the last article, which is given in Chapter 4, the thermal and mechanical performance of GRCs, which are recognized for their enhanced mechanical strength and energy absorption capacity, was studied in the presence of microencapsulated PCM over different service temperatures. Finally, the dissertation was rounded out by the conclusions drawn from the studies and recommendations for future works.

The first study named *"A Taguchi Approach for Optimizing the Mixture Design of Cold Bonded PCM Aggregates"* which is given in Chapter 2, relates to **RQ 1.1** and

RQ 1.2. This is a two-part study in which the first part explains the design, optimization and performance of cold bonded lightweight aggregates. By-products such as FA, GGBS were utilized to manufacture these aggregates. Taguchi method was employed in the experimental design of the aggregate composition. Factors influencing the engineering properties of aggregates and their contribution ratios were investigated using analysis of variance (ANOVA). Optimal values of the factors were determined and in the second part of the research, aggregate mixture design was enhanced using a microencapsulated PCM for a better thermal performance. The effect of different PCM ratios to the engineering properties of the produced pellets was studied experimentally.

The second article named *"A sustainable cold bonded lightweight PCM aggregate production: Its effects on concrete properties"* can be considered as the continuation of the second part of the first study. The article, given in Chapter 3, relates to **RQ 1.1**, **RQ 1.2** and **RQ 1.3**. PCM aggregates, which were designed in the first article using GGBS binder, were manufactured in a way that (i) only cement binded, (ii) using FA and cement, (iii) using GGBS and cement. Effect of binder material composition and PCM amount to the production process and physical, mechanical and thermal properties of produced aggregates were investigated. With different mix designs containing by-products, eco-friendly materials were produced supporting the sustainable development strategies serving the efficient use of energy. Performance of lightweight aggregate concretes (LACs) comprising these PCM aggregates was also investigated to observe the efficiency of the aggregates in concrete.

Third article named *"Performance of Glass Fiber-Reinforced Cement Composites Containing Phase Change Materials"* which is given in Chapter 4, relates to **RQ 2.1**, **RQ 2.2** and **RQ 2.3**. In this study, glass fiber-reinforced cementitious composites were modified using a commercial microencapsulated PCM to improve their thermal performance. The thermal and the mechanical performance of the composites were investigated considering the effect of varying service temperatures which remains on either side of the phase change temperature value of the used PCM.

2. A TAGUCHI APPROACH FOR OPTIMIZING THE MIXTURE DESIGN OF COLD BONDED PCM AGGREGATES[1]

Abstract

Energy efficiency and waste recovery have vital importance for a sustainable environmental progress. This study contributes to both issues considering three different aspects which are (i) production of an energy efficient building material, (ii) utilization of waste materials inside the produced composites and (iii) use of an eco-friendly production method. The first part of the research explains the design, optimization and performance of cold bonded lightweight aggregates. By-products such as fly ash (FA), ground granulated blast furnace slag (GGBS) were utilized to manufacture these aggregates. Taguchi method was employed in the experimental design of the aggregate composition. Factors influencing the engineering properties of aggregates and their contribution ratios were investigated using analysis of variance (ANOVA). Binder composition was found to be the most important factor influencing physical properties, while the curing time was the dominant factor effecting the strength of the aggregates. Optimal values of the factors were determined and in the second part of the research, aggregate mixture design was enhanced using a microencapsulated phase change material (PCM) for a better thermal performance. The effect of different PCM ratios to the engineering properties of the produced pellets was studied experimentally.

Keywords: lightweight aggregate, pelletization method, fly ash, Taguchi method, phase change materials (PCM), energy efficiency

[1] This chapter is based on the paper "Tuncel, E.Y., Pekmezci, B. Y. (2019). A Taguchi Approach for Optimizing the Mixture Design of Cold Bonded PCM Aggregates. *Energy Sources, Part A: Recovery, Utilization, and Environmental Effects.* doi:10.1080/15567036.2019.1697769"

2.1 Introduction

Disposal of waste materials is one of the most prominent environmental problems. Fly ash (FA) and ground granulated blast furnace slag (GGBS) are the industrial by-products which have a serious global production potential, especially in developing countries like Turkey. In 2012, the annual FA production of Turkey was 19 million tons which was 3% of the world production. By 2020, it is expected that this amount will increase by 30%, which corresponds to 25 tons of FA [34]. Total blast furnace slag production of Turkey was reported as 2.7 million tons by 2014 [11]. These products are mostly used in the cement sector, but also frequently encountered as a mineral additive during concrete production [12]. Nevertheless, the utilized portion accounts only for a small proportion of the total material. Another promising way to reclaim these residuals is by artificial aggregate manufacturing [13]. The recovery of by-products has become an essential goal in the mining of natural aggregates, considering the rigorous environmental restrictions imposed on the process in various parts of the world. Utilization of these by-products also enables the conservation of natural aggregate resources and prevention of the damage caused by aggregate mining. Moreover, it is possible to produce lightweight aggregates (LWAs) by artificial manufacturing. When lightweight aggregates are used in construction, dead loads can be decreased by production of smaller structural elements. It enables larger spans for use. Decreased element size also leads to reductions both in cement dosages and reinforcement. Transportation and handling expenses of lighter precast elements will be less. Use of lightweight aggregate also provides advantages like elevated thermal insulation and improved fire resistance [14].

Production of artificial LWAs can be broken down into four basic steps: (i) mixing, (ii) agglomeration, (iii) hardening and (iv) post-treatment. In the first step, raw materials tailored for a specific purpose are mixed with water and other additives. This is a critical step since the constituent materials determine the engineering properties of the aggregate. The agglomeration process, which can be achieved by either granulation or compaction, follows the mixing. Granular aggregates which are also called “green pellets” form in this stage. For the hardening of green pellets, four different methods with various temperature levels exist. The temperature selection depends on the type of binding of the raw materials in LWA. These processes can be seen in Table 2.1

combined with the binding mechanisms. In the cold bonding which is the employed method in this study, binding of the fines is provided via formation of calcium alumina silicate hydrates in a similar manner to regular concrete composites [12]. For this reason, a cementitious binder or a pozzolanic reaction is required for matrix constitution. It is a widely-used method [35], [36], [37], [38], [39] which is prominent because it is environmentally friendly and energy efficient [13], [35], [36], [40]. The last production step involves crushing, sieving and curing, after which the product takes its final form that fits the initial purpose.

Energy efficiency and sustainability are key factors that need to be taken into account while designing new industrial processes. These factors affect the socioeconomic development of countries, along with the life quality [41], [42]. In a world in which energy consumption continues to increase, a universal access to affordable, reliable, sustainable and modern energy has become an essential sustainable development goal [1], [43], [44]. Overexploitation of the limited energy resources and fossil fuel-based pollution are the other environmental concerns promoted by overconsumption. In such a conjuncture, the search for clean and renewable energy alternatives becomes more significant for the countries with external dependence on energy. Turkey is one of the countries that suffer from high expenses of imported energy. For developing countries in which the urban renewal efforts are already in progress, it is imperative to emphasize the energy efficiency and to take precautions to reduce energy costs in new constructions. The building sector reaches up to 40% of the world energy consumption and one third of global greenhouse gas (GHG) emissions is associated with this sector [8]. Furthermore, operational emissions are higher than embodied emissions [9]. Approximately 60% of the energy consumed in buildings is associated with climatization operations such as heating, ventilating and air conditioning (HVAC). Passive cooling strategy is one of the alternatives that can lower the energy used in building heating and cooling. In passive cooling, the energy used for HVAC operations is procured from the external environment and renewable energy sources. Total efficiency of a building can be enhanced by using the passive techniques along with the conventional HVACs. The burden of a conventional system could be minimized with such a strategy [45], [46]. Passive buildings have specially designed structural elements to collect sunlight during the day. They store the energy in their body and return it to the building environment when the temperature drops below a certain level

[15]. PCMs, substances with relatively high latent heat of fusion, can be utilized in building elements such as walls, windows and roofing to take advantage of the solar power. Unlike the old bulky buildings whose large scale carcasses protect the building from excessive thermal fluctuations, buildings containing PCMs offer superior thermal performance with smaller building components owing to their high thermal density. For example, the thermal energy contained in a 25-mm PCM-containing wall is same as that of a 420-mm concrete wall [46], [47]. This allows the prefabrication of the building elements having superior thermal performance in prefabric production plants and very fast installation in situ. PCMs became the focus of many studies due to their three main characteristics. PCMs have relatively steady phase transformation temperature. It provides a stable temperature to the environment during the transformation. Secondly, the wide range of PCM categories (organic, inorganic and eutectic) with several options of phase transformation temperature gives a chance to implementer to choose the appropriate PCM according to application purpose and ambient features. Finally, PCMs are materials offering high latent heat capacity and it is possible to store a large amount of energy in relatively small volumes [48], [49]. Different techniques have been used for the utilization of PCMs in cementitious composites. Encapsulation [50], [51], [52], [53], [54], [55], direct incorporation, immersion [56], [57], [58], [59], [60] and preparing form-stable composite PCMs [61], [62], [63], [64], [65], [66], [67], [68], [69], [70] are the most commonly used methods. There are also a number of review studies in the literature, which explain briefly the types, forms, and characteristics of PCMs [18], [71], [72].

Table 2.1 : Classification of LWA hardening processes based on temperature indication for inorganic matrices [12].

Production process	Temperature (°C)	Binding
Cold bonding	0-100	Matrix with calcium alumina silicate hydrates
Hydrothermal	100-250	Matrix with calcium alumina silicate hydrates
Sintering	1100-1200	Material bridge bonding with alumina silicates
Complete melting	>1200	Alumina silicates

This study explains the production and performance of artificial lightweight aggregates that utilizes waste materials such as FA and GGBS. PCM incorporation to the aggregates is also discussed. It is aimed to contribute to sustainable development of construction sector developing innovative materials which help efficient use of energy and waste recovery. The effects of (i) binder type, (ii) sand/total binder (S/B)

ratio and (iii) curing time to the engineering properties of pellets were investigated experimentally. Taguchi approach was used in the experimental design and optimum factor levels were determined within the region of experimentation. In consideration of the obtained results, the project was carried on with the PCM inclusion to the aggregate mix design. The effect of PCMs to the engineering properties of aggregates were studied by producing different aggregate sets that include various PCM proportions. The physical, mechanical and thermal properties of the produced PCM aggregates were determined.

2.2 Experimental Design and Taguchi's Parameter Design Approach

In engineering, experimental design methods are used for numerous applications [73], [74]. Some potential areas of use:

1. Process troubleshooting
2. Process development and optimization
3. Evaluation of material alternatives
4. Reliability and life testing
5. Performance testing
6. Product design configuration
7. Component tolerance determination

The use experimental design methods allow the mentioned problems to be solved efficiently during the early stages of a product cycle. This approach dramatically lowers the product cost and the time needed for an additional improvement process [75].

Taguchi methods have been widely used for the optimization of products or processes in various engineering areas. The main idea in the method is the reduction of variability around a target (or nominal) value. This target value represents the expected quality characteristics of a product (or process). Any deviation of this nominal is a loss to the consumer or society [76]. Taguchi's robust design approach aims to improve the quality of a product by minimizing the effects of variation without eliminating the causes. Robust design can be described as "an engineering methodology for improving

productivity during research and development so that high-quality products can be produced quickly and at low cost" [77].

Taguchi methodology consists of four basic parts: (i) planning, (ii) conducting, (iii) analysis, and (iv) implementation. The steps involved in each phase can be seen in Figure 2.1 [78]. At the beginning of the process, the quality characteristics to be optimized must be defined. A quality characteristic is a parameter whose variation has a critical effect on the product quality. It is the output or the response variable to be observed. Noise factors are either uncontrollable or too expensive to control parameters. They include variations in environmental operating conditions, deterioration of components with usage, and variations in responses of the same products with the same input. The control parameters are parameters which are thought to have significant effects on the quality characteristics [79]. These factors can be controlled during the production and experimentation.

An optimal experimental design should provide the maximum amount of information after the minimum number of experimental trials. Taguchi Methods serve this purpose by utilizing orthogonal arrays to study a large number of variables with a small number of experiments. An orthogonal array (OA) is a type of experiment where the columns for the independent variables are "orthogonal" to one another. It is defined by (i) number of factors to be studied, (ii) levels for each factor, (iii) the specific 2-factor interactions to be estimated and (iv) the special difficulties that would be encountered during the experiment. Since OAs are highly fractionated factorial designs, using Taguchi approach in experimental design reduces the number of experiments required. This provides several advantages such as: reduction on the experimental effort, time and cost [80].

The analyze of the results is implemented using signal-to-noise (S/N) ratios in Taguchi methods. S/N ratio is a statistical measure of performance expressed in a decibel scale which is derived from the quadratic loss function. One of the following three S/N ratios may be selected depending on the quality characteristics to be optimized:

- smaller-the-better: to be used when the response variable is to be minimized.

$$SN_S = -10\log\left(\frac{1}{n}\sum_{i=1}^{n} y_i^2\right) \tag{2.1}$$

- larger-the-better: to be used then the response variable is to be maximized.

$$SN_S = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \tag{2.2}$$

- nominal-the-best: to be used when a target value is chased for the response variable.

$$SN_T = 10log\left(\frac{\bar{y}^2}{S^2}\right) \tag{2.3}$$

where $\bar{y}$ is mean response (2.4) and S is standard deviation (2.5).

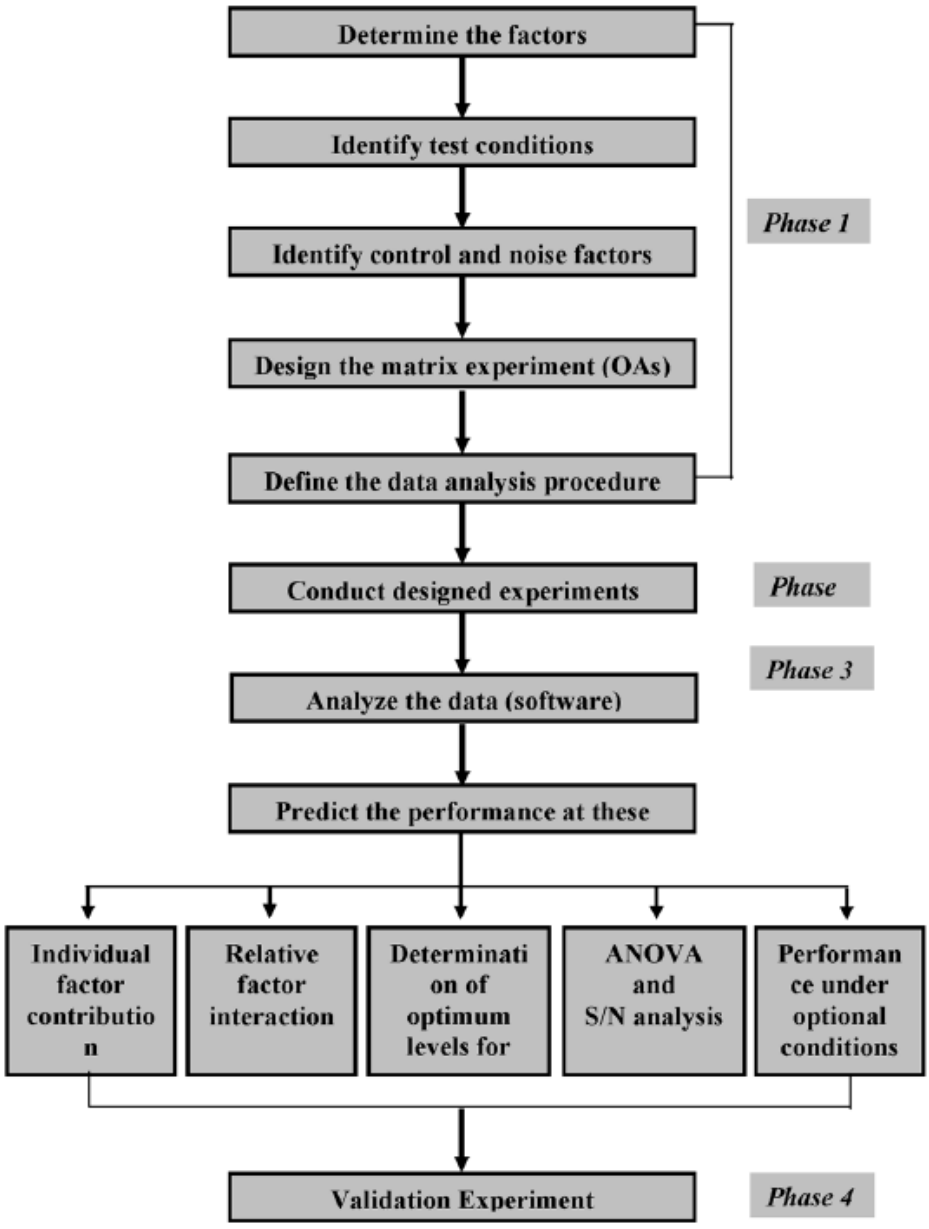


Figure 2.1 : Steps of the systematic approach for Taguchi methodology [78].

$$\bar{y} = \frac{1}{n}\sum_{i=1}^{n} y_i \tag{2.4}$$

$$S = \sqrt{\sum_{i=1}^{n} \frac{(y_i - \bar{y})^2}{n-1}} \tag{2.5}$$

The analysis of the S/N ratio is the same for all of three cases. The factor levels that maximize the S/N ratio are designated as optimal. As a final step, a confirmation test should be done using the predicted optimum levels for the control parameters being studied [77], [79].

2.3 Experimental Study

2.3.1 Materials

Cement (C), Fly Ash (FA), Ground Granulated Blast Furnace Slag (GGBS), natural silica sand and a commercial microencapsulated PCM were used in this study. Cement was an ordinary Portland cement (TS EN 197-1 CEM I - 42.5 R) produced in Turkey. Physical and mechanical properties of the Portland cement are presented in Table 2.2. The fly ash was F type with a density of 2.23 g/ cm^3. GGBS was supplied from Eregli Iron and Steel Plant, Turkey and its density was 2.86 g/cm^3. The chemical analysis of the cement, FA and GGBS is introduced in Table 2.3. Density of the natural silica sand was 2.64 g/cm^3. Particle size distribution of the sand can be seen in Table 2.4.

Table 2.2 : Physical and mechanical properties of the Portland cement.

Density (g/cm^3)	3.15	Specific surface – Blaine (cm^2/gr)	3942
Setting time start (min)	129	Compressive strength (2 days)* (MPa)	29.6
Setting time finish (min)	191	Compressive strength (7 days)* (MPa)	45.8
Volume expansion (Le Chatelier) (mm)	1	Compressive strength (28 days)* (MPa)	56.4

*Average test results of the prism samples with dimension of 40x40x160mm produced using 1-part cement, 3-parts CEN ref. sand, 0.5 water/cement ratio.

The PCM used in this study was a paraffin based powder-type microencapsulated PCM (Micronal DS 5040 X) with a bulk density of 300 kg/m^3. The purpose of the PCM use was to manufacture a cold bonded aggregate which is modified in terms of thermal properties. Because raw PCM induces workability problems through the pelletization

process, microencapsulated PCM was preferred for the work. Besides, paraffin based PCMs are prominent materials for the applications containing cement because of their stability in alkaline environment and resistance in the high pH mediums associated with concrete [30]. Figure 2.2 provides a photograph of the PCM used in the mixtures. The properties of PCM (provided by the manufacturer) are presented in Table 2.5 [81].

Table 2.3 : Chemical composition of cement, fly ash and blast furnace slag.

Chemical component	Cement	Fly ash	Blast furnace slag
SiO_2 (%)	20.14	58.58	42.03
Al_2O_3 (%)	5.04	23.40	10.48
Fe_2O_3 (%)	3.78	6.97	1.19
CaO (%)	63.92	1.55	36.85
MgO (%)	1.35	2.76	5.45
SO_3 (%)	2.84	0.45	0.08
K_2O (%)	0.84	4.11	1.15
Na_2O (%)	0.47	0.46	0.63
Cl (%)	0.0407	0.0319	0.0191

Table 2.4 : Particle size distribution of the natural sand.

Particle size, mm	**4**	**2**	**1**	**0.5**	**0.25**	**0.125**
% fine	100.0	100.0	99.7	99.0	56.9	0.6

Figure 2.2 : Microencapsulated PCM used in mixture.

2.3.2 Design of experiments

Taguchi method was utilized for the experimental design. The mechanical and physical performance of the pellets depend on many different parameters such as components,

the production process and curing conditions. In this study, binder type, sand/binder (S/B) ratio (by weight) and curing duration were selected as factors. Factors and their levels are shown in Table 2.6. As seen from Table 2.6, each factor has three levels. L27 (33) orthogonal array was used to consider the factor interactions. 27 trials were manufactured and tested for mechanical and physical performance (Table 2.7). For physical properties, only binder type and S/B ratio were considered as factors and L9 orthogonal array was used. Contribution percentage of parameters were determined using analysis of variance (ANOVA).

Table 2.5 : Properties of PCM.

Product type	Powder
Particle size (approx. in μm)	50-300
Melting point (approx. in °C)	23
Overall storage capacity (approx. in kJ/kg)	136
Latent heat capacity (approx. in kJ/kg)	96

Table 2.6 : Factors used in the study and their levels.

Factor	Level 1	Level 2	Level 3
A, binder type	cement (100%)	fly ash (70%) + cement (30%)	slag (70%) + cement (30%)
B, S/B ratio	0 (sand-free)	1/3	1/2
C, curing duration (days)	28	56	112

2.3.3 Mixture details and production of the aggregates

The first parameter (Factor A) considered in the study is the binder type (Table 2.6). Three different materials were selected as binder material in aggregate design (Cement, FA and GGBS). While the entire binding material of the aggregate was cement for Level 1, the main binding materials for Level 2 and Level 3 designs were FA and GGBS, respectively. For Level 2 and Level 3 designs, cement was also used as 30% of total binder amount to enable the pozzolanic activity of these minerals. This amount was determined by trial productions, taking into account that cold bonding is the only binding mechanism and no extra strength gaining operations such as sintering or hydrothermal treatments will be applied to the aggregates. In all mixtures that contains mineral admixtures, cement/total binder ratio was kept constant as 30%. Mix designs of the aggregates are given in Table 2.8 where C represents the mixtures which containing only cement as binder (Factor A, Level 1); FA represents the mixtures consisting of fly ash and cement (Factor A, Level 2); S represents the mixtures

consisting of GGBS and cement (Factor A, Level 3). Effect of sand amount on the mechanical and physical properties of the aggregates were also investigated (Factor B). Sand amount was determined in proportion to the amount of binder in the mixture as 0, 1/3 and 1/2 (Table 2.6). In the first column of Table 2.7, the numbers 1, 2, 3 that stand on the right side of the letters in the mixture codes, indicate these proportions, respectively. Water amount was adjusted according to the water need of the mixtures to form regular fresh pellets.

Table 2.7 : A three-level (L_{27}) orthogonal array.

Trial number	Factors and their levels		
	A, binder type	B, S/B ratio	C, curing duration
1 (C1)	1	1	1
2 (C1)	1	1	2
3 (C1)	1	1	3
4 (C2)	1	2	1
5 (C2)	1	2	2
6 (C2)	1	2	3
7 (C3)	1	3	1
8 (C3)	1	3	2
9 (C3)	1	3	3
10 (FA1)	2	1	1
11 (FA1)	2	1	2
12 (FA1)	2	1	3
13 (FA2)	2	2	1
14 (FA2)	2	2	2
15 (FA2)	2	2	3
16 (FA3)	2	3	1
17 (FA3)	2	3	2
18 (FA3)	2	3	3
19 (S1)	3	1	1
20 (S1)	3	1	2
21 (S1)	3	1	3
22 (S2)	3	2	1
23 (S2)	3	2	2
24 (S2)	3	2	3
25 (S3)	3	3	1
26 (S3)	3	3	2
27 (S3)	3	3	3

At the next stage of the study, GGBS aggregates were thermally enhanced using PCM. Two different PCM ratios (6.0% and 12.5% of total dry mixture) were selected considering the ability of the mixture to be pelletized in the disk. Material amounts used in these mixtures are shown at last few rows in Table 2.8 where S represents

PCM-free mixture, which is designed based on S2, and S6PCM and S12.5PCM represent the mixtures containing 6% and 12.5% PCM respectively. The decision of choosing S2 for further modification was made under the guidance of the objectives of the study, some observations during the production and the statistical considerations are described in detail in the following sections.

Table 2.8 : Details of the mixture designs.

Aggregate Group	Code	Cement, g	Fly ash, g	Slag, g	Sand, g	PCM, g	Water, g	Additional mix, g
Group C	C1	400	0	0	0	0	100	150
	C2	300	0	0	100	0	94	150
	C3	267	0	0	133	0	81	150
Group FA	FA1	120	280	0	0	0	106	150
	FA2	90	210	0	100	0	88	150
	FA3	80	187	0	133	0	78	150
Group S	S1	120	0	280	0	0	145	150
	S2	90	0	210	100	0	112	150
	S3	80	0	187	133	0	107	150
Group S/PCM	S	90	0	210	100	0	125	150
(PCM incorporated	S6PCM	85	0	197.4	94	24	137	150
samples)	S12.5PCM	79	0	184	87	50	141	150

A rotating palletization disk seen in Figure 2.3 was used to produce aggregates. It was a single blade disc with a depth of 15 cm and 40 cm in diameter. Parameters such as operation angle (43°) and revolution speed (45 rpm) of the disc was selected based on the findings of a previous study [82]. After a minute of dry mixing, water was added until pellets was formed in the disk. A spray nozzle was used for water feeding. The time between sprays was set to 1 minute. When green pellets were appeared in disk, 150 grams of dry mixture was added to the disk to prevent nascent pellets from growing enormously. The content of this additional mixture is identical to original mixture initially put into the pelletizing disc and the amount was kept constant at 150 grams in all mixtures. Only in mixtures containing PCM, this phase was composed as PCM-free by using only binder, sand and water to create a second phase acting like a shell. With this arrangement, we aimed to minimize possible PCM leaks from the aggregate.

Pellets were cured at 38°C until test day inside a nylon-zipped bag. A wet fabric was also placed into the nylon bag to ensure enough moisture to pellets. Figure 2.4 shows the appearance of the aggregates before the experiment.

Figure 2.3 : Pelletization disk.

Figure 2.4 : The view of the pellets prior to test.

2.3.4 Testing procedure

Physical properties of aggregates by means of oven-dried particle density (ρ_{rd}), saturated and surface-dried particle density (ρ_{ssd}), water absorption capacity by weight (WA) and permeable porosity (PP) were determined using the pycnometer method given in EN 1097-6 standard [83]. Permeable porosity was calculated as the percentage of the water absorption per unit volume. Crushing strength test was employed to determine mechanical performance of the produced aggregate pellets. In this test, a single pellet placed between two parallel steel plates (Figure 2.5(a)) was loaded diametrically until the fracture (split) of the specimen occurred as shown in Figure 2.5 (b). A closed-loop testing machine (INSTRON 5500R) was used in tests. Tensile strength of the individual pellet (MPa), referred to as "crushing strength" (σ_{crush}), was calculated based on the equation 2.6 [13], [84], [85]:

$$\sigma_{crush} = \frac{2.8 \times P_{max}}{\pi \times d^2} \quad (2.6)$$

where, P_{max} is the crushing load representing the maximum force in N that the specimen experienced during the test and d is the distance between loading points given in mm. Curing duration of specimens was chosen as 28, 56 and 112 days for a better hydration of samples containing fly ash and slag (Table 6, Factor C). Ten individual specimens with a diameter of 9.0 ± 1.0 mm were tested from each group. Since size distribution of the aggregates containing PCM was relatively finer, tests were conducted on specimens with a diameter of 6.6 ± 1.0 mm.

(a) (b)

Figure 2.5 : Crushing stress test (a) and test specimens after fracture (b).

Thermal properties of PCM and aggregates containing PCM (Group S6PCM and S12.5PCM) were determined using Heat-Flux Differential Scanning Calorimetry (DSC) method. A control group which has no PCM inside was also tested (Group S). DSC tests were conducted using a Shimadzu DSC-60A Differential Scanning Calorimeter under a nitrogen atmosphere. Temperature range of the calorimeter is −140 to 600°C and its calorimetric measurement range is ±150 mW. DSC test samples were prepared by crushing the aggregate pellets into small particles. Sample mass was approximately 70 mg. The selected temperature range to study the thermal transitions of the PCM was -5°C to 45°C. The dynamic scan was started by cooling from 40 to −10°C and continued with dynamic heating segments that had heating or cooling rates of 5°C/min. The test procedure was repeated three times for every composite type, with virgin samples each time. The phase change temperatures and solidification-melting enthalpies were obtained from the heat flow measured during the tests.

2.4 Results and Discussion

2.4.1 Effect of mixture composition on manufacture process

When the pelletization process was repeated with different ingredients, the observations during the manufacture showed that the mixture ingredients affected agglomeration tendency. The pellet formation became easier as the amount of sand in the mixture increased. However, when the amount of sand in the mixture passed a certain threshold, the amount of binder in the mixture became insufficient to bind the whole content. This led to the accumulation of unbound material in the base of the pelletization disk. After a series of production trials, the range of the sand proportions to be used in the study was determined.

Another point to be noted is the variation of the water needed to form green pellets. As seen in Table 2.8, the amount of water used in the process decreases as the sand amount increases. This can be explained by the reduction of the total amount of fine material in the mixture. In all mixtures, the total amount of the dry material entering the process was kept constant. This means that any increase in the amount of sand will result in a reduction in the amount of binding material which have a relatively fine composition in reference to the sand. In terms of workability, as the water requirement of the fine materials would be higher, the increase in the sand ratio in the mixture caused a decrease in the water requirement of the mixtures. Binder ratio of mixture also decreases with the inclusion of sand. This resulted in an increase in the water/binder ratio up to 3%. The water need was also varied depending on the binder composition. In fly ash-containing mixtures, more water was needed compared to the mixtures using only cement as a binder, whereas the highest water requirement was observed in the slag mixtures. In summary, the need for water increases as the grain size distribution gets finer.

PCMs extended the duration of the process and increased the water amount used in pelletization. In the blends containing more than 12.5% PCM, the pellets began to get flatten and flat aggregate granules appeared instead of the spherical pellets. Hydrophobic nature of PCM inhibited the formation of a thin liquid film on fine particles which is necessary for agglomeration according to the theory of pelletization [82]. The pellet formation was accomplished by increasing the moisture content of

mixture, but the excessive water emergent in use of high PCM rates caused mixture to sludge. So, the PCM amount was limited to 12.5% for a stable production (Table 2.8).

2.4.2 Physical properties of pelletized aggregates

Physical properties of aggregates are given in Table 2.9. Density (oven dried) of aggregates changed between 1.36 and 2.22 g/cm^3. When these values evaluated considering the related European Standard, aggregates manufactured using FA and S can be classified as LWA [86]. FA use caused the aggregates to be lighter approximately 10% in comparison with fully cement used ones. This ratio increased to 22% in slag use. In the case of PCM use, decline in density reached to 41%. In other words, by the use of GGBS and PCM, the density of the artificial aggregates is reduced by almost half. The density increased when the sand ratio was increased in the mixtures. This phenomenon can be explained by the change in the aggregate pore structure. As described in the previous section, the water requirement of the mixtures decreased with the increase of the sand ratio in the mixture. Reduced water amount in the mixture means less capillary gaps per unit volume. This is also supported by the permeable porosity values given in Table 2.9. In this table, it is seen that porosity values of mixtures containing sand are lower. As a result, the increase in aggregate compacity has brought about an increase in density. The maximum increase was observed in slag containing mixtures as 9%. It can be explained by the difference between the density of sand and other ingredients. The optimum level for each factor with factor impact order were determined by Taguchi Analysis (Table 2.10). According to S/N ratios and mean test results presented in Table 10, the optimum conditions for density was A3B1 which corresponds to S1 mixture. Figure 2.6 shows effects of factor levels on mean results. The most effective parameter on density was determined to be the binder type with a contribution percentage of 93.5 (Table 2.11). It must be noted that the PCM incorporated samples were prepared after these analyses, these mixtures were not included in statistical analysis. When the mixtures containing PCM are examined, it was observed that the use of 6% PCM reduced the density from 1.67 g/cm^3 to 1.49 g/cm^3 which corresponds a 11% decline. %12.5 PCM use decreased the density to 1.36 g/cm^3 corresponding a 18.5% decline. This shows that PCM, which was used in aggregates for thermal enhancement, also contributed the lightness of the aggregates.

Table 2.9 : Physical properties of pelletized aggregates.

Aggregate type	Oven-dried particle density (g/cm^3)	Saturated and surface-dried particle density (g/cm^3)	Water absorption (WA) by weight (%)	Permeable porosity (PP) (%)
	ρ_{rd}	ρ_{ssd}	WA	PP
C1	2.22	2.38	7	16
C2	2.28	2.39	5	11
C3	2.31	2.42	5	12
FA1	1.98	2.18	10	20
FA2	2.06	2.20	7	14
FA3	2.08	2.23	7	16
S1	1.67	1.87	12	20
S2	1.83	2.00	9	17
S3	1.82	2.00	10	18
S	1.67	1,96	17	28
S6PCM	1.49	1,78	19	29
S12.5PCM	1.36	1,65	21	29

Table 2.10 : Mean S/N ratios and mean test results for each level of each factor.

Factors	Mean S/N Ratios (dB) / Mean of Means		
	Level 1	Level 2	Level 3
Density (g/cm^3)			
A[a]	-7.12 / 2.27	-6.19 / 2.04	-4.97 / 1.77*
B[b]	-5.77 / 1.96*	-6.23 / 2.06	-6.28 / 2.07
Water absorption (%)			
A[a]	-14.98 / 5.7*	-18.11 / 8.2	-20.21 / 10.3
B[b]	-19.59 / 9.7	-16.61 / 7.0*	-17.1 / 7.4
Permeable porosity (%)			
A[a]	-22.14 / 13.0*	-24.26 / 16.5	-25.19 / 18.2
B[b]	-25.35 / 18.6	-22.88 / 14.1*	-23.37 / 15.0
Crushing strength (MPa)			
A[b]	20.97 / 11.4*	19.39 / 10.4	18.21 / 8.2
B[c]	19.12 / 9.6	19.77 / 10.3*	19.69 / 10.1
C[a]	16.86 / 7.2	19.94 / 10.0	21.78 / 12.7*

* Optimum level.

[a] Most effective factor.

[b] 2nd effective factor.

[c] 3rd effective factor.

PP as well WA capacity of aggregates increased when FA and S were incorporated into the production. Binder type was determined as the most effective factor (Table 2.10). Since binder type affected the amount of water used in production, it also caused pore structure of the aggregates to change. In parallel with the increase of porosity, the water absorption capacities of the samples increased. The contribution of binder type

to the WA and PP was found as 70.5% and 54.5% respectively (Table 2.12 - Table 2.13). Use of sand decreased the WA and PP while PCM incorporation increased them.

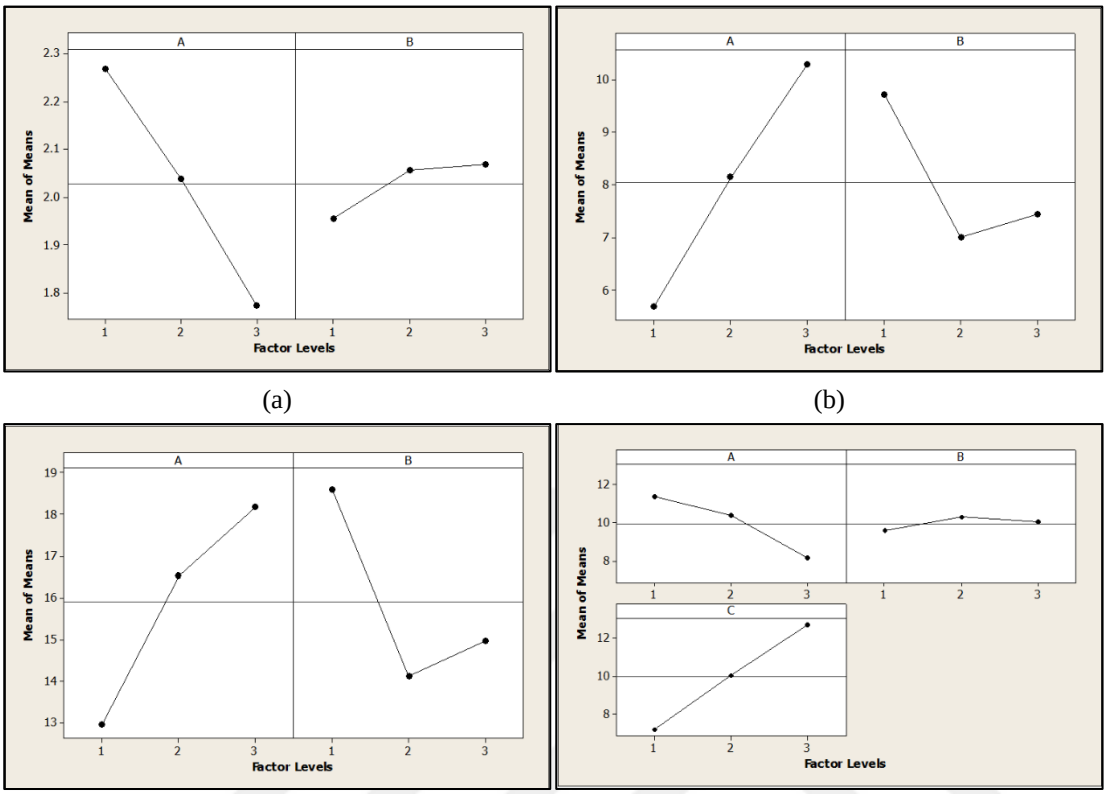


Figure 2.6 : Main effects plot for means (a) Density (b) Water absorption (c) Permeable porosity (d) Crushing strength.

Table 2.11 : Results of ANOVA for density.

Factor	d_f	SS	V_A	F	Contribution (%)
A, binder type	2	0.37069	0.1853	260.64	93.5
B, S/B ratio	2	0.02302	0.0115	16.19	5.8
Error	4	0.00284	0.0007		0.7
Total	8	0.39656			100.0

Table 2.12 : Results of ANOVA for water absorption.

Factor	d_f	SS	V_A	F	Contribution (%)
A, binder type	2	31.7956	15.8978	168.33	70.5
B, S/B ratio	2	12.9489	6.4744	68.55	28.7
Error	4	0.3778	0.0944		0.8
Total	8	45.1222			100.0

Table 2.13 : Results of ANOVA for permeable porosity.

Factor	d_f	SS	V_A	F	Contribution (%)
A, binder type	2	44.6670	22.3333	33.50	54.5
B, S/B ratio	2	34.6670	17.3333	26.00	42.3
Error	4	2.6670	0.6667		3.3
Total	8	82.0000			100.0

2.4.3 Crushing strength of pelletized aggregates

Figure 2.7 shows the average of crushing strength values of pelletized aggregates. Maximum strength of aggregates was 17.7 MPa at the end of the 112 days while the minimum value was obtained as 8.2 MPa for PCM-free mixtures. According to the results of ANOVA (Table 2.14), the most significant parameter affecting the crushing strength is the curing duration of samples with a 50.6% contribution. The interaction of the binder type and the curing duration (A*C) was also highly effective on strength (26.6% contribution). The reason for this is that the FA and GGBS used in the mixtures are pozzolanic materials and the curing time becomes a significant parameter in the case of pozzolan use. The effect of S/B ratio on strength was found to be insignificant with a contribution of less than 1%. Main effect plots for crushing strength are given in Figure 2.6 (d). In Table 10 and Figure 2.6 (d), the optimum level for the binder type appears as Level 1, which represents aggregates where only cement is used as a binder (Group C). Although Group C had a better performance at 28th day, Group FA caught up Group C at 56th day and passed it at 112th day. This can be explained by the pozzolanic character of FA that shows up in later ages with continuous curing. Aggregates in which GGBS was used along with cement showed a better early strength performance in comparison with FA containing ones. The optimum factor levels were determined to be A1B2C3 (Table 2.10) in the end of Taguchi analysis.

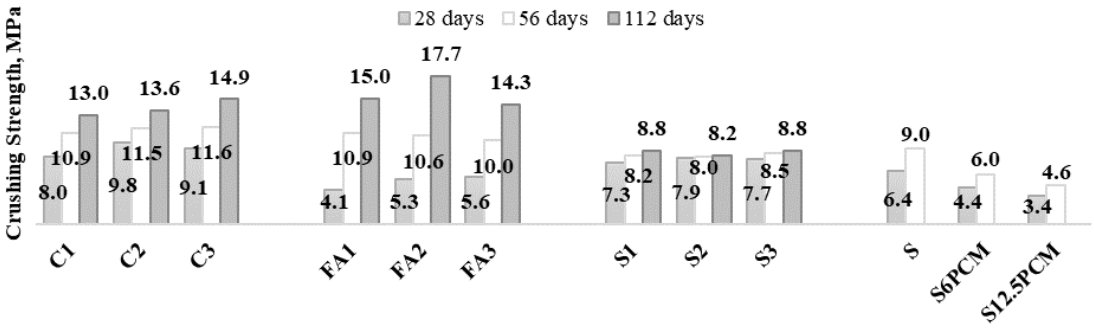


Figure 2.7 : Crushing strengths of aggregates.

PCM incorporation decreased the mechanical performance of aggregates. 6% PCM use decreased the 56-days strength from 9 MPa to 6 MPa which corresponds to a 33.3% decline. In the case of 12.5% PCM use, crushing strength of aggregates dropped 49% to 4.6 MPa. In Figure 2.8, a micrograph of S12.5PCM is shown. Although PCM distribution over the cross section was uniform, PCM particles approaching each other at higher usage ratios may disconnect the continuous cementitious matrix and result a

decrease in strength. This phenomenon is seen better in scanning electron microscopy (SEM) image given in Figure 2.9 which shows an individual microencapsulated PCM particle in the S12.5PCM specimen and damaged adjacent particles. The porous structure of PCM particles is also seen in this figure.

Table 2.14 : Results of ANOVA for crushing strength.

Factor	d_f	SS	V_A	F	Contribution (%)
A, binder type	2	49.0450	24.5226	35.82	18.2
B, S/B ratio	2	2.3650	1.1826	1.73	0.9
C, curing duration	2	136.1560	68.0781	99.45	50.6
A*B	4	3.3210	0.8304	1.21	1.2
A*C	4	71.5300	17.8826	26.12	26.6
B*C	4	1.3370	0.3343	0.49	0.5
Error	8	5.4760	0.6846		2.0
Total	26	269.2300			100.0

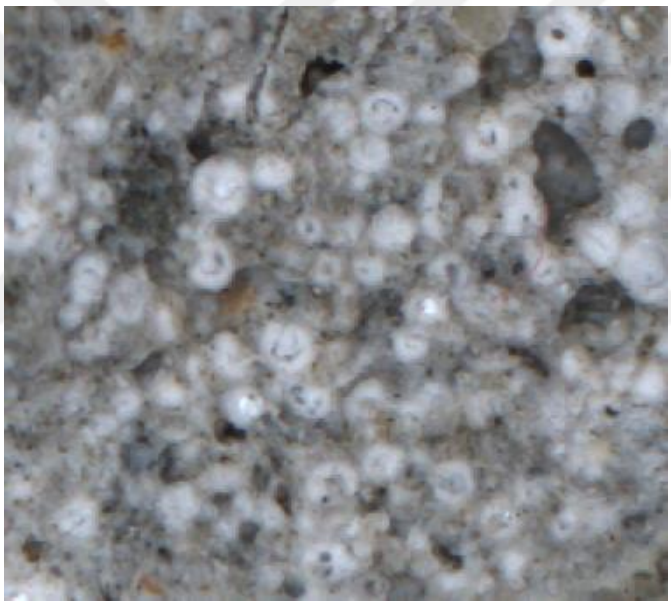


Figure 2.8 : Micrograph of S12.5PCM.

2.4.4 Thermal properties of pelletized aggregates

In the thermal analysis of both PCM and pelletized PCM aggregates, DSC method was used. In DSC tests, the heat flow to the specimen was analyzed with respect to the reference. The difference gives the heat absorbed and released during melting and solidification and thermogram which is obtained after the test introduces properties such as specific heat, phase change temperature and latent heat. In Table 2.15, some of these characteristic values obtained from DSC curves are presented. In the table, T_{onset} stands for the temperature value at which phase transition begins, while T_{endset} is the temperature value at which the phase transition is over. T_{peak} represents the

temperature value at which the DSC curve changes direction (inflection point). These three points are also important in terms of the calculation of phase change enthalpies of melting and solidification process, which are indicated as ΔH_{endoth} and ΔH_{exoth} in the table, respectively. Detailed information about method, calculation and DSC curves of the PCM used in the study can be found in a previous work of the authors [87].

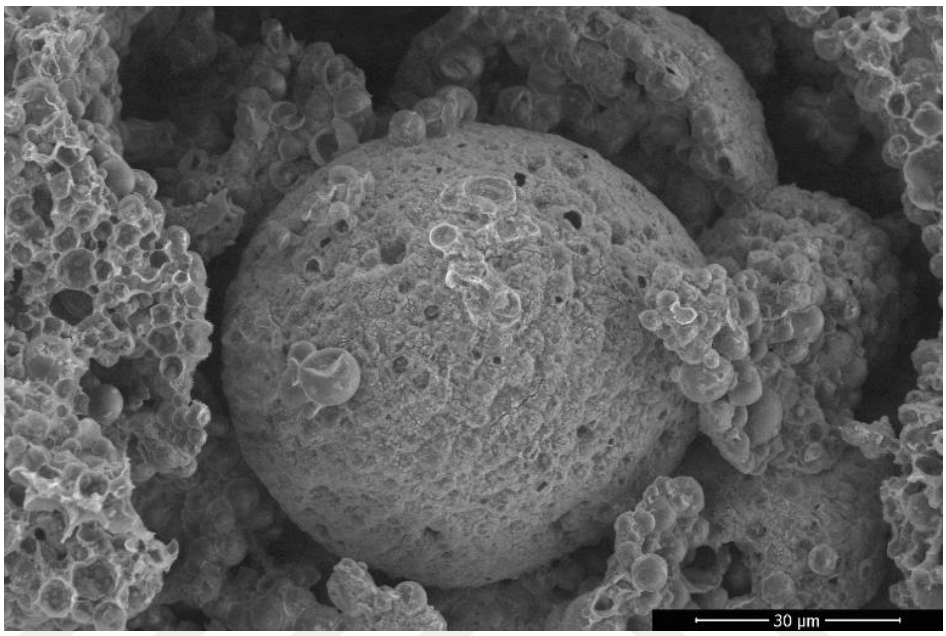


Figure 2.9 : Internal structure of S12.5PCM focused an individual PCM microencapsule.

At heating session, melting of the PCM started at 21.05℃ and process ended at 31.40℃. These values are important to indicate the operating range of the PCM. Considering that the PCM will be used in building elements such as walls, windows and roofing, when the temperature of the building elements reaches 21℃, the PCM in the building materials will begin to melt and the phase transition process will continue up to 31.4℃. During this transition, PCM will reduce the load of HVAC systems by preventing the solar based temperature rise inside the building. The enthalpy of melting was calculated as 83.95 kJ/kg. At cooling, PCM started to solidify at 22.83℃ and phase transition finished at 10.87℃. This means when the sunset begins and temperature starts to drop, the PCM in the building material will begin to solidify at 22.83℃ and the phase change transition will last up to 10.87℃. Within this period, PCM will return the solar energy stored to the environment and the temperature change in the building will occur in a softer manner. This will also help the HVAC system in the building by reducing the burden on the system. The enthalpy for this transition was calculated to be 91.32 kJ/kg which was 95% of the value declared by the manufacturer

(Table 2.5). Different transition temperature ranges at melting and solidification are a phenomenon previously observed in the literature and this difference in between the start and end point of transition at melting and solidification may be attributed relatively higher values of the heating or cooling rate [88], [89].

Table 2.15 : Thermal properties of PCM used in the study and manufactured LWAs.

Mixture Code	PCM Amount (%)	Melting Process				Solidification Process			
		T_{peak} (°C)	T_{onset} (°C)	T_{endset} (°C)	ΔH_{endoth} (kJ/kg)	T_{peak} (°C)	T_{onset} (°C)	T_{endset} (°C)	ΔH_{exoth} (kJ/kg)
PCM	100	25.54	21.05	31.40	83.95	18.22	22.83	10.67	91.32
S	0	-	-	-	-	-	-	-	-
S6PCM	6	24.06	19.55	25.73	1.59	17.11	20.03	10.45	2.31
S12.5PCM	12.5	23.06	20.02	24.86	2.56	18.34	20.89	14.34	2.66

When the results are reviewed, it is seen that onset, endset and peak temperature obtained in aggregate tests are coherent with those of the PCM. The reason of this harmony is that the PCM in aggregates is the only content that undergoes phase transition in the exposed temperature range. As seen from Table 2.15, phase transition of the aggregates started at 20°C in melting process and completed at 25°C after making a peak at 24°C. It started at 20°C an ended in between 10°C - 14°C with a peak of 18°C in solidification. For a reasonable indoor comfort, these values reveal a functional performance. When the enthalpy values of the aggregates are scrutinized, it is seen that these values are fairly low for an effective energy storage and they are considerably below the enthalpy value of pure PCM. For a better thermal performance, aggregates with higher PCM ratios may be produced as long as the strength reduction caused by PCM use is kept under control. Additional comments on the subject are included in the discussion section.

2.4.5 Optimization based on Taguchi Methodology

The aim of the process optimization is to figure out the optimal values of the factors inside of the region of experimentation that maximize the concerned property [73]. In this study, concerned result properties such as density, porosity, crushing strength are more than one. When Table 2.10 is reviewed, it is seen that the optimum factor levels for different properties may conflict with each other. For example, the first level of Factor A is the optimum level for water absorption and porosity while the third level is the optimum level for density. In such a case, it would be more appropriate to select the optimum conditions according to the intended use of the product. In this study,

because it is aimed to produce as much lightweight aggregates as possible by using waste materials, the optimum level for Factor A is determined as Level 3. This corresponds to the mixture which contains GGBS as binder. For Factor B, which represents the sand / binder ratio of the mixtures, the second level is determined as the optimum level because of the observations during the pelleting process together with the fact that Level 2 is the optimum level for the water absorption and permeable porosity properties (Table 2.10). The most favorable condition for Factor C, which represents the curing duration of the aggregates, is the condition that stands for the highest curing time, as for other composites containing cement. In this study, the optimum level for Factor C is determined as Level 3.

The last phase of Taguchi approach is the validation experiments using the predicted optimum levels for the control parameters being studied. In the second part of the study, when the mixture design of PCM containing aggregates were determined, the optimum factor levels obtained in the previous stage of the research were used. The PCM free mixture (S) that belongs the S/PCM aggregate group (Table 2.8) is the mixture that uses the optimum factor levels and test results related to this mixture can be evaluated as confirmation results. In Table 2.16, these results are summarized. For comparison, the results of S2 mixture are also given in the table. It must be noted that the curing duration is 56 days for these tests. When the strength values of the GGBS containing aggregate group (S1, S2, S3) are reviewed (Fig. 7), it is seen that the difference between 56-day strength and 112-day strength is relatively low when the waiting period is considered. Therefore, in order to respond quicly to a possible sectoral demand for produced aggregate and to ensure product continuity in practice, 56-day standard strengths were considered to be sufficient and 56-day curing period was adopted for the next stage of the study.

Table 2.16 : Optimal values of the factors and results of the validation tests.

Aggregate Type	Factor Levels			Experimental Results				
	A	B	C	ρ_{rd} (g/cm^3)	ρ_{ssd} (g/cm^3)	WA (%)	PP (%)	σ_{crush} (MPa)
S2	3	2	2	1.83	2.00	9	17	8.0
S (Validation)	3	2	2	1.67	1,96	17	28	9.0

When the results of the confirmation tests are compared with the former results (Table 2.16), it is seen that aggregate density values are lower while WA and PP are higher. This is because the higher water amount of this production (Table 2.8). The excess water used caused the aggregate structure to be more porous and the corresponding

values changed slightly. Despite the increasing amount of water, an increase in aggregate strength values is observed. This is due to the decrease in the specimen size used in the crushing strength tests. It should be noted that the crushing strength of the pellets increases as the particle diameter decreases [82].

2.5 Discussion

In this experimental study, artificial aggregates utilizing industrial by-products and phase change materials were designed and produced for use in concrete. Artificial aggregate production is important in terms of recycling waste materials, protection of natural aggregate resources and prevention of mining damages for a sustainable environmental development. On the other hand, it gives us an opportunity to manufacture lightweight aggregates leading lighter concrete designs. It must be noted that implementation of the pelletization process is the key to the desired results because it directly affects the properties of the produced aggregates. When the results of the study were evaluated together with the literature, it was observed that the strength of the produced aggregates were higher than the previous studies [82], [90], [91]. The main reason of this is the relatively higher cement amounts of the aggregates. Cement triggered the pozzolanic activities of FA and GGBS inside aggregates and it also provides an extra binding feature to the mixtures. Density of the aggregates also changed depending on the cement amount (Table 2.9). Although the fully cement-bonded group showed the best strength performance of the study, they remained outside of the LWA classification [86]. In this context, it can be said that mixtures containing FA and GGBS yielded more optimum results. GGBS bonded group showed better early strength results comparing the FA group (Figure 2.7) and their density values was lower. In contrast, the final strength of the FA aggregates was approximately twice that of the GGBS aggregates. The most important reason for this is the higher water / binder ratio of GGBS aggregates compared to other groups due to the need for more water in the pelletization process. GGBS group was opted for the next stage of study because of its lightness and rapid strength gaining and its thermal performance was improved using PCM. Microencapsulated PCM was used due to the fact that raw PCM prevents agglomeration of the fines with its sticky nature. Although the phase transition temperature range of produced aggregates was determined to be consistent with pure PCM, the energy stored in aggregate was obtained significantly

lower than that of the literature-cited macroencapsulated PCM aggregates which were produced by impregnation of liquid PCM into LWAs [92], [93], [94]. In addition to the different methodology, different PCM types used in those studies and their phase change enthalpy values brought this result. On the other hand, Sharifi et al. reported some problems of absorption method such as the interference of PCMs with hydration reactions and sticking PCM portions on the surface of the carriers or leakage of PCM which causes unwanted effects to different properties of the binder [95]. With use of microencapsulated PCM, the possible interactions between PCM and other materials used in production are minimized. Another point to be addressed regarding this issue is the damage done to PCM capsules during DSC enthalphy measurement test period. When testing the thermal properties of microencapsulated PCM-containing aggregates, the original aggregate structure is deformed during the sample preparation as required for test method. PCM capsules within the aggregate were also damaged. In DSC test preparation, sticky nature of grinded aggregate particles encountered during the crushing process of the PCM-containing aggregates supports this idea. As a result, the enthalpy values calculated based on the experimental results might be different from the actual situation and may remain below the actual potential of the aggregates. On the other hand, micro scaled samples measured in mg level, decreases the chance of making a homogenous sampling that represents overeall aggregate, especially in relatively low amounts of PCM containing mixtures. It is therefore difficult to take consistent results with DSC test of composite aggregate that reflect macro properties. Within the limits of this study, although no quantitative comparable results could be obtained with the DSC tests, higher latent heats were observed with the increasing PCM amounts in the aggregate. As future work, LWAs with high porosity may be produced by pelletization method and after a desired strength development, a better thermal performance can be obtained by PCM impregnation method. In this way, sintering or hydrothermal treatment can be applied to aggregates to provide them extra strength prior to the PCM impregnation process. As suggested by Cui et al., aggregate surfaces may be coated with epoxy or modified cement paste to prevent PCM leakage during phase change process [94].

Another important issue about the PCM-integrated building materials is the economic feasibility of the materials produced. As the PCMs, especially the microencapsulated ones, are materials with higher costs comparing to the other components of

cementitious composites, the economic aspect of the use of these materials become a key concern in practice. Using PCM at low levels and choosing relatively untreated PCM may be effective for limiting the initial cost of the resulting PCM composites. The compatibility between PCM and other matrix materials must also be considered. The main point here is that by virtue of the energy efficiency characteristics redounded to the buildings by PCM usage, initial investments can be compensated at acceptable times [96], [97], [98]. This compensation times would decrease with decrease of initial price of PCM in the future. To enhance the system efficiency, PCM phase transition temperatures must be selected considering the climate zone of the structure to be built [87], [96]. Gas tariffs, electricity tariffs, capital cost of PCMs, and bank interest are also important for the economic assessment of the PCM utilization [87] and must be evaluated in the design phase of the project.

2.6 Conclusion

Cold bonded lightweight aggregates were produced using cement, fly ash, slag and sand. Physical properties and mechanical performance of the aggregates were investigated and evaluated by using Taguchi method. It was observed that the use of sand facilitates pelleting. Binder type is more effective on physical properties than S/B ratio according to Taguchi analysis. When GGBS was used as binder, lighter aggregates were obtained compared to C and FA binder ones. ANOVA results showed that S/B ratio has a significant effect on porosity of aggregates. The effect of binder type, S/B ratio and curing duration on crushing strength of aggregates were studied and the most significant factor was obtained as curing time. Since FA and GGBS are pozzolanic materials, aggregate strength showed significant changes with extended curing period according to the type of binder used. It was obtained that the use of GGBS allows the production of lightweight aggregates with sufficient early strength. By incorporating a microencapsulated PCM into the production process, lighter and thermally enhanced aggregates were produced in the second stage of the research. Sand was also used in design of PCM aggregates because it helps to pellet formation. Although it was below the expected level, PCM addition improved the thermal capacity of aggregates. However, a significant decrease in strength with the increase of PCM amount was observed. It can be concluded that as a future work, an optimization can be done targeting effective and efficient PCM use.

3. A SUSTAINABLE COLD BONDED LIGHTWEIGHT PCM AGGREGATE PRODUCTION: ITS EFFECTS ON CONCRETE PROPERTIES[1]

Abstract

Due to the responsibility of building sector for a large portion of the energy consumed, it is necessary to prioritize the energy efficiency along whole lifecycle of a building. Designing buildings to provide maximum benefit from renewable energy sources, and reusing the by-products such as fly ash and blast furnace slag makes a significant difference with regard to sustainability and energy efficiency. In this study, artificial lightweight aggregates (LWAs) were produced utilizing phase change materials (PCMs) which are prominent in literature with their contribution to energy efficiency via their high thermal capacity. Supplementary cementitious materials (SCMs) were utilized as part of binder of LWAs. Pelletization method was used in production of LWAs. Effect of binder material composition and PCM amount to the production process and physical, mechanical and thermal properties of produced aggregates were investigated. As the amount of PCM increased, lighter aggregates with higher thermal capacities were obtained. The strength decreased significantly with use of PCM. Use of PCM at high amounts negatively affected the pelleting process as well. Concrete samples were produced using LWAs and tested to determine the effect of aggregates to concrete performance. The minimum compressive strength obtained was 33.9 MPa, which is accepted as satisfactory for many structural applications.

Keywords: phase change materials (PCM), lightweight aggregate (LWA), energy efficiency, pelletization, cold bonded aggregates, PCM pellets

[1] This chapter is based on the paper "Tuncel, E.Y., Pekmezci, B. Y. (2019). A Sustainable Cold Bonded Lightweight PCM Aggregate Production: Its Effects on Concrete Properties. *Construction and Building Materials, 181,* 199–216."

3.1 Introduction

Sustainability has long been a universal development goal in many fronts. Sustainable development is improvement of the present by prioritizing next generations' ability to meet their own needs, calls for concerted efforts for an inclusive, sustainable and resilient future. Universal access to affordable, reliable, sustainable and modern energy and efficient use of it is one of the sustainable development goals declared by the United Nations (UN) in the 2030 Agenda for Sustainable Development. Energy production and use are also closely related to several other goals determined by UN and they form the basis of a more sustainable world plan [1], [99]. On the other hand, energy consumption of the world has been increasing dramatically during the last century resulting a depletion in energy costs and environment degradation. This increase has reached over 30% in the last two decades [4], [43], [44], [100], [101]. Moreover, as projected by the U.S. Energy Information Administration (EIA), the demand in energy will continue increasing progressively through the forthcoming thirty years and total world energy consumption will rise from 549 quadrillion Btu in 2012 to 815 quadrillion Btu in 2040, with an increase of 48% [6]. The current energy demand has mainly been supplied by fossil fuels, which are well-known with their contribution to the greenhouse effect, promoting global warming. Besides, the world is running out of fossil fuels and the supply is not economically reliable [4], [5]. Building sector accounts for around 40% of world's energy consumption and one third of associated global greenhouse gas (GHG) emissions [8]. This consumption and emission are notable in all stages of a building lifecycle from construction to demolition. Furthermore, it is known that operational emissions are higher than embodied emissions [9]. Therefore, limitation of the building based energy consumption by improving the energy efficiency of both new and refurbished buildings, remains at the forefront of the governmental plans and building codes in order to ensure a sustainable development strategy [10].

Considering that approximately 60% of the energy consumed in buildings is associated with climatization operations such as heating, ventilating and air conditioning (HVAC), lowering the energy used for these purposes are essential for sustainable buildings concepts. Passive cooling strategy is an alternative method in building climatization which makes construction material work as natural climatization agent.

In this concept, there is no energy input except for renewable energy sources. Unlike the traditional HVAC systems, in passive cooling, the energy available in nature is utilized instead of conventional energy resources. It is also possible to enhance the total efficiency of the building by using the passive techniques along with the conventional HVACs, reducing the burden of them. Such applications are called ‘‘hybrid” systems in literature [45], [46]. Phase change materials (PCMs) which have considerable latent heat capacity are very advisable components for passive buildings. It is possible to store energy or to control the indoor temperature fluctuations (to the extent permitted by the phase transition range of the chosen PCM) with the use of PCMs [72], [102]. When the environmental temperature rises above the PCM’s melting point, PCM transitions from solid to liquid by absorbing the excessive heat and it solidifies upon a drop in the temperature while releasing the heat to the indoor. PCM remains at an almost constant temperature during the transition and prevents overheating or overcooling of the interior [4], [103]. Integration of the PCM into the building fabric provides enhanced thermal storage effects and improved thermal comfort. Although PCMs can be integrated with almost all kinds and components of building envelopes, the PCM integration in wallboards, roof & ceiling, and windows is most commonly studied, due to its convenience and efficiency [45].

Concrete can be considered for utilization of PCM since it is the most widely used construction material. It is a practical material with its design indulgence, ease of production and testability. More importantly, concrete offers large areas for heat exchange with smaller heat exchange depth which promotes performance of the PCM [92]. It is reported that PCM use may help to minimize thermal cracking of massive concrete sections and to reduce the freeze/thaw damage [56], [104]. Different techniques, such as direct incorporation, immersion [56], [57], [58], [59], [60], and encapsulation [50], [51], [52], [53], [54], [55] have been used for the utilization of PCM in cementitious composites. Direct incorporation and immersion technics are addressed in the literature for their practicability and economy but leakage possibility of PCM after large number of thermal cycling makes these methods ineffective. Besides, the leaked PCM may interfere with hydration products and can cause mechanical and durability problems [92]. For the case of microencapsulation technique, desired PCMs with an appropriate melting point is enclosed in microscopically small polymer capsules and then incorporated in building materials

like plasterboards, mortars or concrete. Waxes can be used as PCM to obtain melting points that can be flexibly adjusted to the application. The encapsulation process protects the wax from any deterioration and wax can stay in its pure form with its high heat storage capacity.

Although the microencapsulation method was preferred by many researchers [105], [106], [107], problems like leakage of PCM from damaged micro capsules still continue to be a matter of debate [88]. Macro encapsulation methods have been proposed to address this problem [51], [93], [108], [109] but extensive studies addressing the effect of PCM use to the mechanical performance of concrete are needed to add on the current findings [92]. In this project, we utilize microencapsulated PCM with some by-products like fly ash and blast furnace slag to develop a cementitious lightweight PCM aggregate using pelletization method. By these ready-to-use PCM aggregates, structural concretes with improved thermal properties can be made more accessible.

Pelletization is a frequently used method [12], [13], [35], [36], [37], [110], [111], [112], [113] for production of artificial aggregates. In this method, the fines are agglomerated in a rotating disc or drum with the help of moisture and an additional binder if the need arises. Pelletization can be practiced via cold bonding or thermal treatment methods such as sintering, autoclaving and steam curing may be applied to improve the pellet characteristics. In cold bonding method, pozzolanic ability of ingredients is employed at ambient temperature. It is a preferred procedure of producing artificial aggregate by many researchers [35], [36], [37], [38], [39], [113] due to its energy efficiency [40]. Theory of pelletization can be found in the work by Baykal and Döven [82]. Artificial aggregates may have customized properties and they can be reproduced consistently according to manufacturer purpose. They are also promising materials in terms of ensuring sustainability by allowing the already existing natural aggregate resources to be consumed in a controlled manner. Moreover, the use of waste materials such as fly ash, blast furnace slag or reservoir sediments in aggregate production is both economically and environmentally beneficial [38]. The technical benefits such as lower density of these by-products are also taken.

This paper explains the experimental study carried out for the development of lightweight thermal aggregate (LWTA) products enhanced by microencapsulated PCM. With different mix designs containing by-products such as fly ash and blast

furnace slag, eco-friendly materials were produced supporting the sustainable development strategies with the efficient use of energy. Physical, mechanical and thermal characterization of cold bonded aggregates were studied systematically. Performance of lightweight aggregate concretes (LACs) comprising these PCM aggregates was also investigated to observe the efficiency of LWTA in concrete.

3.2 Experimental Work

3.2.1 Material properties

An ordinary Portland cement (CEM I – 42.5 R) with a density of 3.15 g/cm^3 was used. Physical and mechanical properties of the cement are presented in Table 3.1. The fly ash used in the mixture was F type with a density of 2.23 g/cm^3 and specific surface of 4512 cm^2/g. The next component was the blast furnace slag which was supplied from Eregli Iron and Steel Plant, Turkey with the density of 2.86 g/cm^3 and specific surface of 5128 cm^2/g. The chemical analysis of the cement, fly ash and slag is given in Table 3.2. Natural silica sand was also used in mixtures and its sieve analysis is presented in Table 3.3.

Table 3.1 : Physical and mechanical properties of the Portland cement.

Density (g/cm^3)	3.15	Specific surface – Blaine (cm^2/g)	3942
Setting time start (min)	129	Compressive strength (2 days)* (MPa)	29.6
Setting time finish (min)	191	Compressive strength (7 days)* (MPa)	45.8
Volume expansion (Le Chatelier) (mm)	1	Compressive strength (28 days)* (MPa)	56.4

*Average test results of the prism samples with dimension of 40x40x160mm produced using 1-part cement, 3-parts CEN ref. sand, 0.5 water/cement ratio

Table 3.2 : Chemical composition of cement, fly ash and blast furnace slag.

Chemical component	Cement	Fly ash	Blast furnace slag
SiO_2 (%)	20.14	58.58	42.03
Al_2O_3 (%)	5.04	23.40	10.48
Fe_2O_3 (%)	3.78	6.97	1.19
CaO (%)	63.92	1.55	36.85
MgO (%)	1.35	2.76	5.45
SO_3 (%)	2.84	0.45	0.08
K_2O (%)	0.84	4.11	1.15
Na_2O (%)	0.47	0.46	0.63
Cl (%)	0.0407	0.0319	0.0191

Table 3.3 : Particle size distribution of the natural sand.

Particle size, mm	**4**	**2**	**1**	**0,5**	**0,25**	**0,125**
% fine	100,0	100,0	99,7	99,0	56,9	0,6

A commercial, paraffin based powder-type microencapsulated PCM (Micronal DS 5040 X) with a bulk density of 300 kg/m^3 was selected to enhance the thermal performance of the aggregates. The reason of this selection is that the paraffin PCMs offer certain advantages such as: (i) high purity, leading very high melting enthalpy, (ii) high resistance to oxygen based degradation risks, (iii) high stability unlike salt hydrates, (iv) the hydrophobic nature, ensuring superior water repellent properties. [114]. Unlike many other PCMs, paraffin based ones are suitable for cementitious applications because they are stable in alkaline medium and resistant to degradation in the high pH environments associated with concrete [30]. Figure 3.1 provides a photograph of the PCM used in the mixtures. The properties of PCM (provided by the manufacturer) are presented in Table 3.4 [81].

Figure 3.1 : Microencapsulated PCM used in mixture.

Table 3.4 : Properties of PCM.

Product type	Powder
Particle size (μm)	50-300
Melting point (approx. in °C)	23
Overall storage capacity (approx. in kJ/kg)	136
Latent heat capacity (approx. in kJ/kg)	96

3.2.2 Methodology for the production of aggregates

Three main aggregate sets were designed containing cement (C), fly ash (FA) and slag (S) and each group were enhanced by the incorporation of PCM in varying amounts. Main groups were entitled as C-LWA, FA-LWA and S-LWA. Mixture design properties can be seen in Table 3.5. In mixture codes, the number, which comes after the group code C, FA or S, indicates the PCM percentage with respect to total dry mixture mass. By dry mixture, the mixture of granular materials which is consisting of cement, SCMs, sand and microencapsulated PCM used in production was expressed. In mixtures coded with C, only cement was used as binder. FA and S samples were blended mixtures of fly ash and slag with cement. Cement ratio to total binder amount was 30% in these mixtures. Sand : total binder ratio of the mixtures was also kept constant as 1:3 by mass in all groups. Different PCM amounts varying between 6% and 50% of total dry mixture by mass were used to evaluate the effect of PCM. Water amount was adjusted according to the water need of the mixtures to form regular fresh pellets. Material proportions respect to the total dry material mass are given in Table 3.6.

Table 3.5 : Mixture details of pelletized aggregates.

Code	Cement, g	Fly ash, g	Slag, g	Sand, g	PCM, g	Water, g	Additional dry mix, g
C	300.0	0	0	100.0	0.0	82.0	150.0
C6PCM	282.0	0	0	94.0	24.0	106.0	150.0
C12.5PCM	262.0	0	0	87.5	50.0	114.0	150.0
C25PCM	225.0	0	0	75.0	100.0	146.0	150.0
C50PCM	150.0	0	0	50.0	200.0	190.0	150.0
FA	90.0	210.0	0	100.0	0.0	95.5	150.0
FA6PCM	84.6	197.4	0	94.0	24.0	117.0	150.0
FA12.5PCM	78.8	183.8	0	87.5	50.0	121.0	150.0
S	90.0	0	210.0	100.0	0.0	125.0	150.0
S6PCM	84.6	0	197.4	94.0	24.0	137.0	150.0
S12.5PCM	78.8	0	183.8	87.5	50.0	141.0	150.0

For the case of aggregate pelletization, a pelletizer disc which is 15cm deep and 40cm in diameter was used. The angle of disc plane to the normal (operation angle) and revolution speed of the disc were adjusted as 43° and 45 rounds per minute (rpm), respectively, based on the findings of a previous study [82]. A photograph of the pelletization disk setup is shown in Figure 3.2 (a). Materials were mixed by hand in a container before they were put into the disk for the pelletization. In Figure 3.2 (b), an

appearance of a PCM integrated mix prior to the pelletization process is presented. After the dry mixture was put into the disk, it was mixed about 1 minute as dry and after that water was added slowly with a spray nozzle. Water amount for each spray was approximately 3 to 5 grams. The time interval was adjusted around 1 minute for sprays. Water feeding was stopped when green pellets started to appear in the disk. When the formed pellets reached an average diameter of 6 mm, 150 grams of dry mixture (referred as "Additional dry mix" in Table 3.5) was added to the disk to prevent the fresh pellets from going too big in size. This additional mixture was composed as PCM-free by using only binder, sand and water. By this way, a second phase acting like a shell was created on the outer side of the pellets and impermeability of the aggregate was enhanced. A possible PCM leak, which could occur if the PCM capsules were damaged, was also avoided. Green pellets are shown in Figure 3.3 (a).

Table 3.6 : Material proportions of the mixture (% of dry mixture).

Main group code	Subgroup code	Cement, %	Fly ash, %	Slag, %	Sand, %	PCM, %
C-LWA	C	75.0	0.0	0.0	25.0	0.0
	C6PCM	70.5	0.0	0.0	23.5	6.0
	C12.5PCM	65.6	0.0	0.0	21.9	12.5
	C25PCM	56.3	0.0	0.0	18.8	25.0
	C50PCM	37.5	0.0	0.0	12.5	50.0
FA-LWA	FA	22.5	52.5	0.0	25.0	0.0
	FA6PCM	21.2	49.4	0.0	23.5	6.0
	FA12.5PCM	19.7	45.9	0.0	21.9	12.5
S-LWA	S	22.5	0.0	52.5	25.0	0.0
	S6PCM	21.2	0.0	49.4	23.5	6.0
	S12.5PCM	19.7	0.0	45.9	21.9	12.5

When the PCM amount of the mixture was increased, the pelletization process took longer and the mixture in the disk tended to sludging without any pellet formation. For the case of 25% and 50% PCM use, angular aggregate pellets with a tender and slender nature were obtained. Because of this tendency, PCM ratio of mixtures was restricted to 12.5% in fly ash and slag groups.

After fresh pellets lost their free surface water, they were cured in nylon-zipped bags with a wet rag to provide moisture for samples and then they were kept in 38°C until test day. A photograph showing the pellets before the experiment can be seen in Figure 3.3 (b).

(a) (b)

Figure 3.2 : Pelletization disk (a) and a PCM compounded mixture prior to pelletization process (b).

(a) (b)

Figure 3.3 : Green pellets (a) and the view of the pellets prior to test (b).

3.2.3 Concrete designs and production

To evaluate the effect of the PCM pellets to concrete, mixtures were produced and tested to reveal mechanical performance. In mix designs, all parameters were kept constant except the artificial aggregate type. Maximum aggregate size was limited up to 8 mm and CEN standard reference sand was used as fine aggregate together with produced pellets. In all mixtures, 55% of the total aggregate amount was preferred as CEN standard sand while 45% of it was the produced artificial aggregate. By this way, a proper particle size distribution consistent with the aggregate reference curves

specified in TS 802 standard was obtained [115]. Particle size distribution of composed aggregate mixes can be seen in Figure 3.4 together with the reference curves. In this figure, each aggregate mix is denoted by related concrete name which is starting with "C/" and containing the artificial aggregate type used in it. A8, B8 and C8 are the curves that determine the recommended granulometry range for the relevant standard. All mixes were prepared in a laboratory mixer with vertical rotation axis by forced mixing. The cement dosage was selected as 500 kg/m^3 and water/cement ratio was kept constant as 0.38 in all mixtures. Details of the mix designs are presented in Table 3.7. 40x40x160 mm prisms were molded and they were cured in lime saturated water at a constant temperature of 23±2 ℃ until tested.

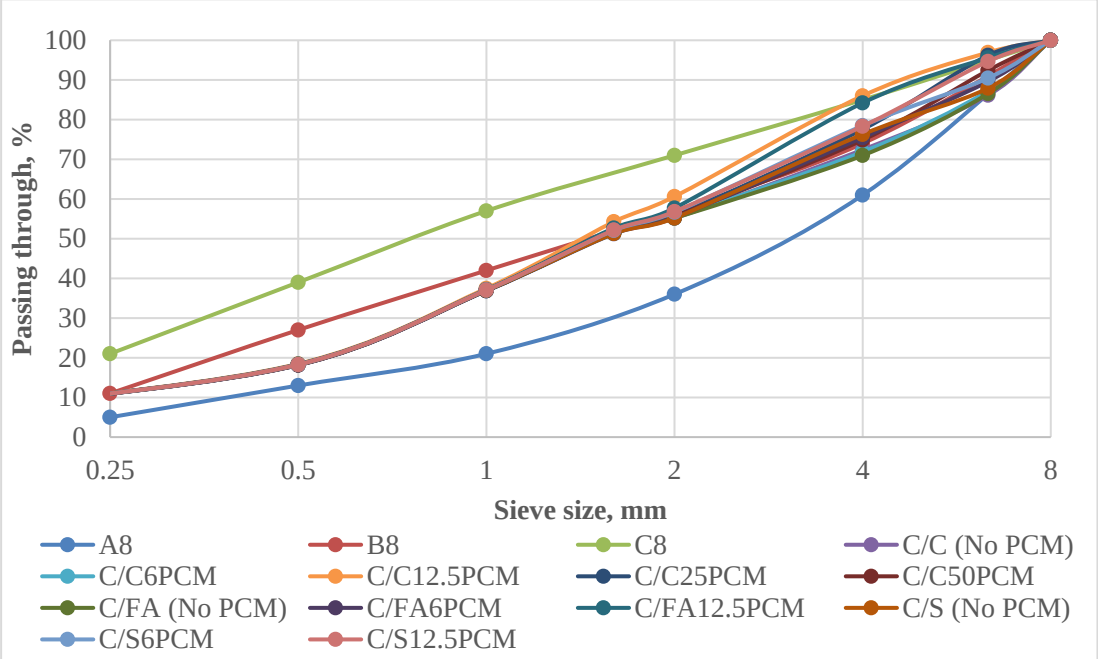


Figure 3.4 : The particle size distribution curves of the aggregate mixes used in concrete with reference to the borders (A8, B8, C8) [115].

3.2.4 Test procedures

After sieving of particles larger than 8 mm, particle size distribution of the aggregates was determined by sieve analysis in accordance with EN 933-1 standard [116]. Physical properties of aggregates including oven-dried particle density (ρ_{rd}), saturated and surface-dried particle density (ρ_{ssd}) and water absorption capacity (WA) were determined using the pycnometer method given in EN 1097-6 standard [83]. Permeable (open) porosity of aggregates was calculated using equation 3.1, based on the concept of water absorption by volume [55], [117], [118]:

$$\text{Permeable porosity (\%)} = \frac{M_1 - M_4}{M_1 - (M_2 - M_3)} \times 100 \quad (3.1)$$

where, M_1 is the mass of the saturated and surface-dried aggregate in the air, in grams; M_2 is the mass of the pycnometer containing the sample of saturated aggregate and water, in grams; M_3 is the mass of the pycnometer filled with water only, in grams; M_4 is the mass of the oven-dried test portion in air, in grams.

Table 3.7 : Concrete mix designs.

Mixture #	Artificial aggregate type used in the mixture	Cement, kg/m^3	Water, kg/m^3	Artificial aggregate, kg/m^3	Sand, kg/m^3	Water/cement ratio
1	C	500	190	711.5	942.1	0.38
2	C6PCM	500	190	611.2	942.1	0.38
3	C12.5PCM	500	190	538.6	942.1	0.38
4	C25PCM	500	190	458.4	942.1	0.38
5	C50PCM	500	190	367.3	942.1	0.38
6	FA	500	190	644.1	942.1	0.38
7	FA6PCM	500	190	565.0	942.1	0.38
8	FA12.5PCM	500	190	528.0	942.1	0.38
9	S	500	190	573.1	942.1	0.38
10	S6PCM	500	190	522.2	942.1	0.38
11	S12.5PCM	500	190	484.3	942.1	0.38

For evaluation of the strength characteristics of the produced LWTAs, crushing strength test was applied on spherical aggregate pellets using a closed-loop testing machine (INSTRON 5500R). In this test, a single pellet placed between two parallel steel plates was loaded diametrically until the fracture (split) of the specimen occurred as shown in Figure 3.5. Tensile strength of the individual pellet (MPa), referred to as "crushing strength" (σ_{crush}), was calculated based on the equation 3.2 [13], [84], [85]:

$$\sigma_{crush} = \frac{2.8 \times P_{max}}{\pi \times d^2} \quad (3.2)$$

where, P_{max} is the crushing load representing the maximum force in N that the specimen experienced during the test, and d is the distance between loading points, given in mm. Tests were performed on 28-day old samples as well as 56-day old samples for a better pozzolanic reaction of samples containing fly ash and slag. Ten individual specimens with a diameter of 6.6 ± 1.0 mm were tested from each group. The diameter range of test samples was decided considering the general size distribution of the pellets together with their fineness modulus. It should be noted that the crushing strength of the pellets decreases as the particle diameter increases [82].

The thermal properties of the PCM and LWTAs were analyzed by Heat-Flux Differential Scanning Calorimetry (DSC). DSC monitors heat energy changes associated with phase transitions and chemical reactions. In this technique, the temperature difference between the sample and a reference is measured as a function of temperature or time, under controlled temperatures. The temperature difference is proportional to the change in the heat flux (energy input per unit time).

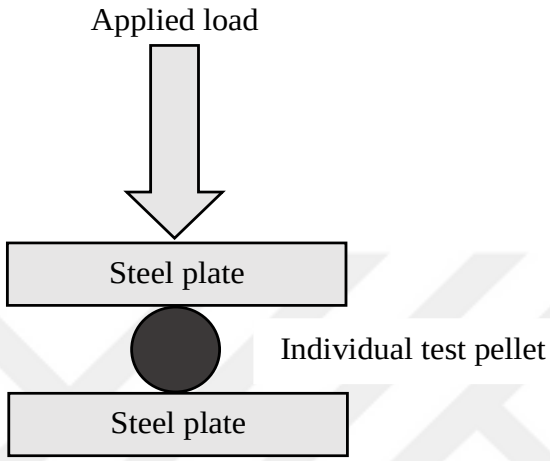


Figure 3.5 : A schematic illustration of crushing strength test.

In a typical heat-flux DSC cell, the sample (contained in a metal pan) and reference (an empty pan) sit on raised platforms formed in a thermoelectric (constantan) disk, which serves as the primary means of heat transfer to the sample and reference from a temperature-programmed furnace (heating block). Traditionally, the temperature of the furnace is raised or lowered in a linear fashion, while the resultant differential heat flow to the sample and reference is monitored by plate thermocouples fixed to the underside of the disk platforms. These thermocouples are connected in series and measure the differential heat flow using the thermal equivalent of Ohm's Law:

$$dQ = dT/R \tag{3.3}$$

where dQ is the difference in heat flow between sample and reference, dT is the temperature difference measured and R is the thermal resistance of the cell [119].

In a typical DSC, after selection of the linear temperature scan rate, the sample heater power is adjusted to keep the sample and reference at the same temperature during scan. The difference in the two input signals is determined by amplifiers and this power difference is recorded automatically and reflected in the DSC curves which provide information such as phase change temperatures, solidification and melting enthalpies.

The enthalpy of the phase transition is calculated from the area under the peaks by integrating the peak above/below the baseline [120], [121].

Reference aggregate groups produced without PCM were also tested in addition to those containing the PCM. DSC test samples were prepared by crushing the aggregate pellets into small particles. DSC tests were conducted using a Shimadzu DSC-60A Differential Scanning Calorimeter under a nitrogen atmosphere. The DSC sample mass was approximately 70 mg. The sealed sample (placed in a platinum pan) was placed in the calorimeter oven together with an empty reference pan. The selected temperature range to study the thermal transitions of the PCM was -5°C to 45°C. The dynamic scan was started by cooling from 45°C to -5°C and continued with dynamic heating segments that had heating or cooling rates of 5°C/min. The test procedure was repeated three times for every composite type, with virgin samples each time. The phase change temperatures and solidification-melting enthalpies were obtained from the heat flow measured during the tests.

Physical properties of the concrete samples containing pelletized LWTAs were determined in accordance with EN 12390-7 standard [122]. Five specimens from each batch were dried in an oven at 105°Cuntil the mass drop has stopped. Volume of specimens were obtained by water displacement method specified in the relevant standard. Water saturated density, oven-dried density, water absorption and permeable porosity of samples were calculated to compare the effect of different aggregate types.

The mechanical performance of the concretes was assessed by means of flexural and compressive tests which were conducted 28 days after the first production. Three-point bending test was performed on three individual prisms for each set. Compressive tests were carried out on halves of the prisms broken at the end of bending tests. Six individual specimens for each group were tested in compression.

Micro structure analyses on the fractured surface of the tested aggregates were conducted through scanning electron microscopy (SEM). Differences between the matrix material and PCM distribution in the aggregates were analyzed. Concrete samples were also examined using optical microscope imagery after cutting transversely.

3.3 Observations, Results and Discussions

3.3.1 Effect of PCM on pelletization process and particle size distribution of LWTAs

The inclusion of PCM in the blends reduced the tendency of fines to agglomerate and the duration of the process was extended. Table 3.8 shows particle size distribution of pelletized aggregates, generated pellets became smaller and the shape of grains started to flatten (Figure 3.6). This phenomenon can be explained by the mechanism of pellet formation along with the hydrophobic nature of the PCM. According to the theory of pelletization, the agglomeration of moisturized fines starts with the formation of a thin, meniscus shaped liquid film on the surface of the grains. This liquid film can be thought of as bridges connecting the grains together. As a result of the centrifugal and gravitational forces generated when the particles are rotated in a disk or drum, the bonding forces between the grains are strengthened and fresh pellets which are called as “green pellets” form [82]. The water repellency of the PCM incorporated into the blends, prevents the formation of thin liquid film on fine particles and makes the formation of a water bridge between two particles difficult. To overcome this problem, the moisture content of the mixture has been increased and start of the pellet formation was initiated. Although pellet formation has been successfully achieved; at the PCM rates of 25% and above, the process became more unstable and the mixture tended to sludge with excessive increase of the water content in the disk. To avoid the sludging, the amount of moisture has been increased with very small increments and the mixture has been given enough time to use the moisture properly. With the intend of a stable production process, the PCM amount was restricted by 12.5% in fly ash and slag aggregates.

Table 3.8 : Particle size distribution of the pelletized aggregates.

Material	Passing, % Sieve Size, mm						
	8	**6.35**	**4**	**2**	**1.6**	**1**	**0.5**
C	100.0	69.2	38.7	0.3	0.2	0.0	0.0
C6PCM	100.0	71.6	37.5	0.9	0.5	0.1	0.1
C12,5PCM	100.0	93.1	68.9	12.5	6.9	1.4	0.7
C25PCM	100.0	91.5	50.1	1.6	0.9	0.1	0.0
C50PCM	100.0	83.1	44.2	1.6	0.9	0.1	0.0
FA	100.0	70.1	35.5	0.4	0.3	0.3	0.2
FA6PCM	100.0	77.0	45.5	0.8	0.5	0.3	0.3
FA12,5PCM	100.0	90.3	64.9	6.0	3.4	0.7	0.5
S	100.0	73.2	47.3	47.3	0.5	0.4	0.3
S6PCM	100.0	78.9	52.2	3.7	2.1	0.4	0.3
S12,5PCM	100.0	88.2	51.8	4.1	2.3	0.5	0.3

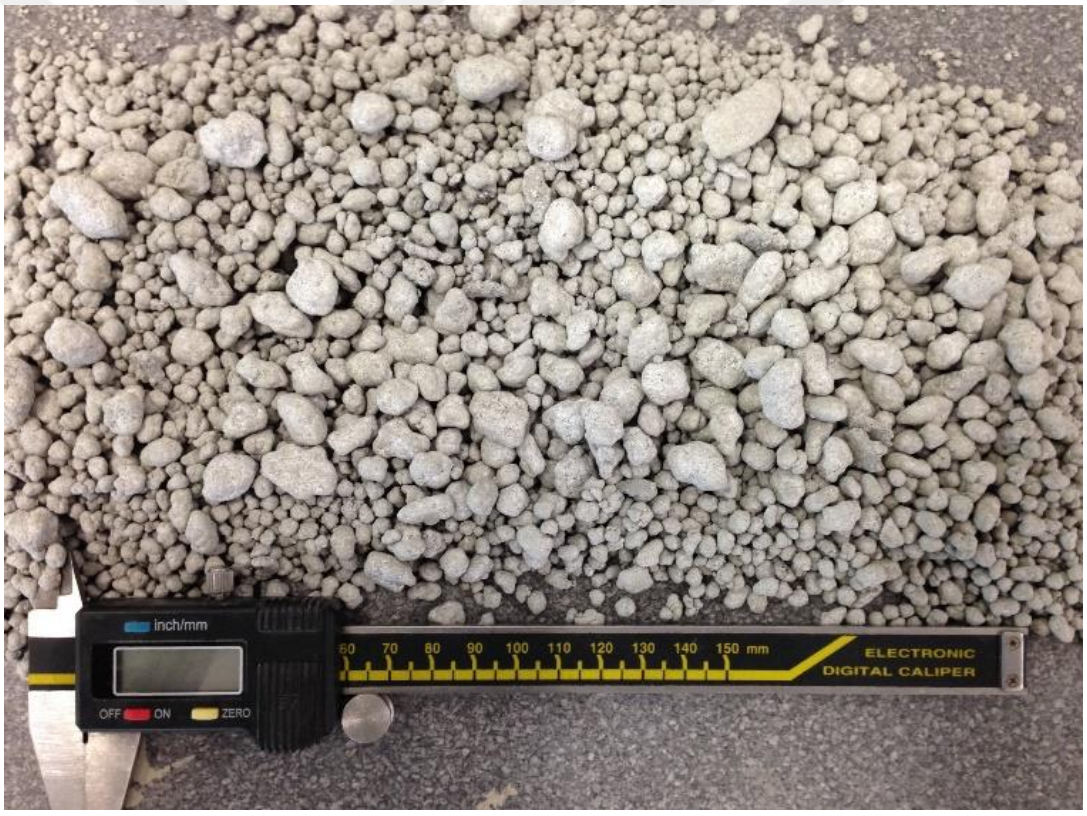


Figure 3.6 : LWTA pellets consisting 50% PCM (C50PCM).

The variation in the amount of water that mixtures need during pelletization process should be emphasized. When mixture details of manufactured aggregates (given in Table 3.5) are investigated, it is seen that the water used to form pellets from fines varies by binder type as well as PCM ratio of the mixture. When the reference mixtures C, S and FA were compared among themselves, the water / binder ratio of the cement based mixture (C), which was 0.20, increased to 0.23 in samples containing fly ash

(FA), while it increased to 0.30 in slag containing samples (S). PCM incorporation also increased the amount of water needed. Reason of this increment can be explained by the increase of fine material amount in unit volume. Supplementary cementitious materials (SCM) and PCM have less unit weight comparing to cement. Since material replacements in designs were made by weight in this study, the fine material content increased in unit volume of the mixture. SCM and PCM have higher specific surface area than cement which is another factor that increased water need. The relation between the water / total binder ratio and PCM ratio of mixture is presented in Figure 3.7. The graph reveals that the water / binder ratio increases in a linear manner with the increase of the PCM ratio, regardless from binder type. Only in S-LWAs, this increase was slighter compared to C-LWAs and FA-LWAs for peer groups in terms of PCM quantity. This reduction in increase ratio can be explained by aforementioned higher water / binder ratio of S-LWA reference sample. The PCM ratio on water demand of aggregates is more significant than the binder type (Figure 3.7).

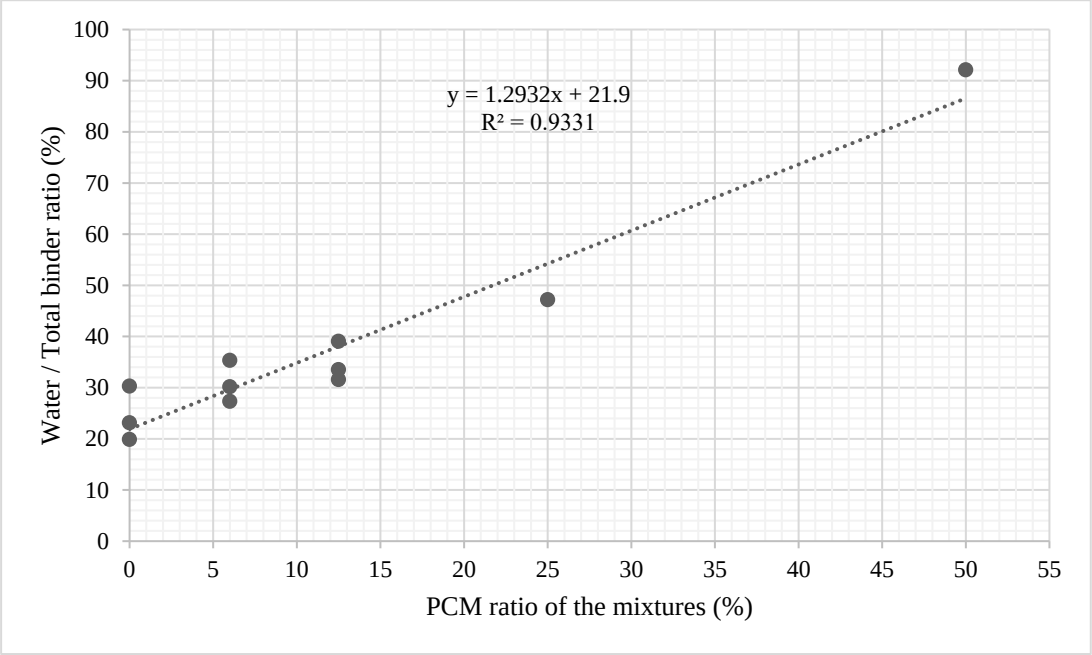


Figure 3.7 : Variation of water / binder ratio with PCM ratio of the aggregate.

3.3.2 SEM analysis on aggregate pellets

SEM analyses have revealed the structural differences between aggregates arising from both binder composition and PCM use. The results of these analyses obtained from the fracture surface of the tested samples are also very important in terms of explaining the different mechanical behaviors of the mixtures. Particle size of PCM

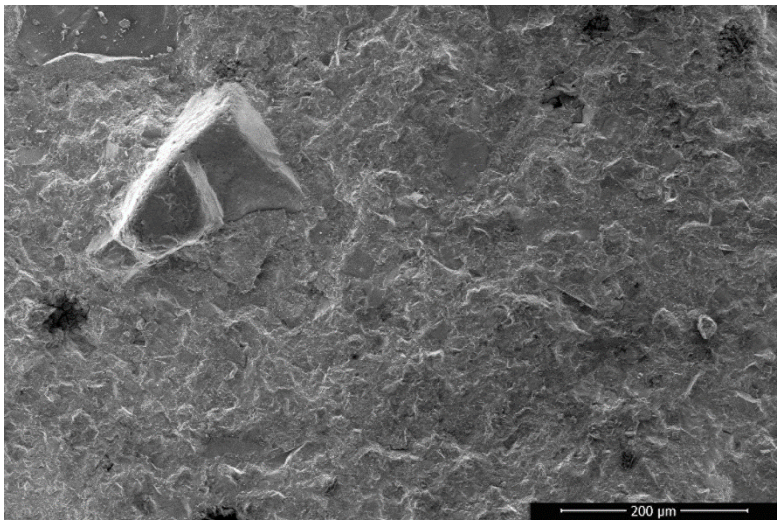
was obtained between 50-300 μm as consistent with the information provided from manufacturer. When the reference samples C, FA and S were analyzed (Figure 3.8), a denser structure stands out in cement based mixture [Figure 3.8 (a)] while S exhibits a porous, weaker look [Figure 3.8 (c)]. In addition to the mix ingredients, differences in the water amount of pellets mentioned in the previous section have also a serious effect on the micro structure. These differences have created different structures influencing to the physical, mechanical and thermal properties of the aggregates.

In Figure 3.9, the change in the micro structure of cement based aggregates with the increase of PCM amount is shown. As the PCM amount in mixture increases, the structure and volume of the matrix material also change. As explained above, the use of PCM, increases the water requirement of the mixture, causing the matrix to weaken. On the other hand, the increase of PCM volume in the mixture causes the PCM particles to approach each other in the structure, causing the matrix phase to decrease in volume gradually. It is clearly seen that when PCM is used at 25% [Figure 3.9 (c)], matrix becomes very weak and in case of 50% use [Figure 3.9 (d)], almost all the fracture surface is formed by PCM particles and matrix becomes invisible.

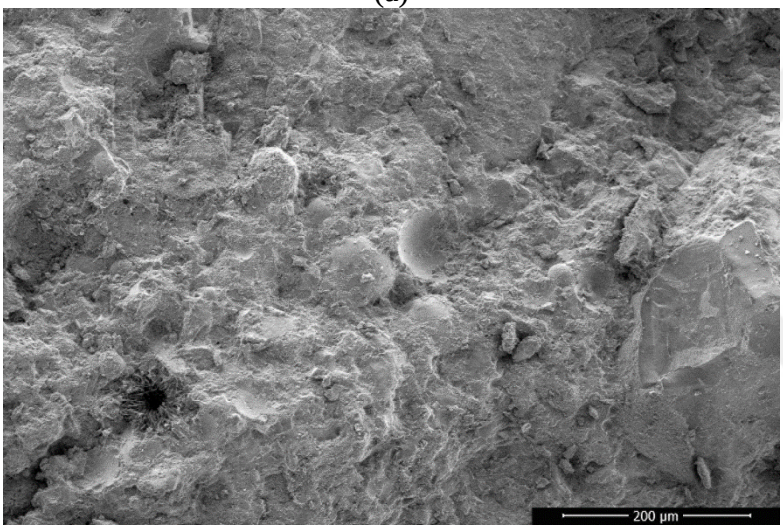
Similar results were obtained in cement and SCM based mixtures which contain PCM and the change of micro structure according to aggregate groups is given in Figure 3.10. It can be concluded that uniform micro structures meaning homogenous distribution of matrix are observed in aggregates up to 12.5% PCM ratio.

3.3.3 Effects of binder type and PCM content on the physical properties of LWTA

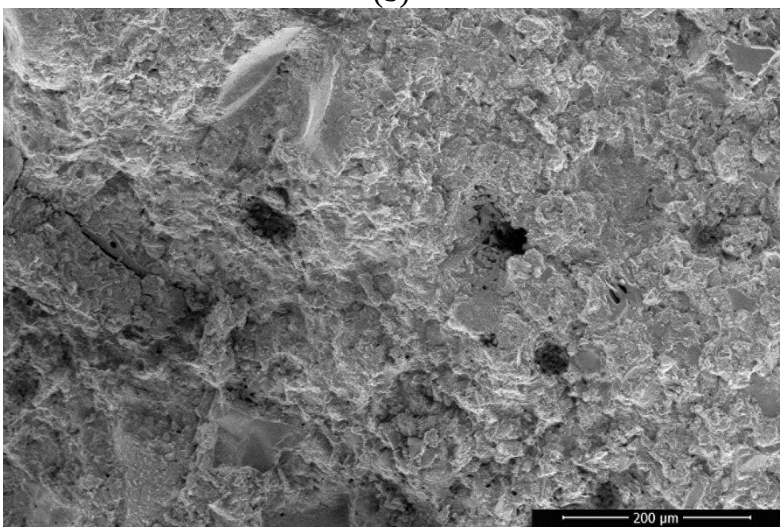
Oven-dried density (ρ_{rd}) and saturated and surface-dried density (ρ_{ssd}) of manufactured aggregates are presented in Figure 3.11. Oven dried density values were between 0.99 and 2.3 g/cm^3. In the European Standard which defines LWA, the limit value of the particle density for LWA is specified as 2.00 g/cm^3 [86]. All mixtures manufactured within this study can be classified as a lightweight aggregate except the reference mixture of C-LWA which does not include any PCM, fly ash or slag. When densities of the reference mixtures (C, FA and S) compared with each other, the lightest one is S with a density of 1.67 gr/cm^3 which is 27.4% lighter than the one consisting totally cement binder. FA lightweight aggregate specimens are also lighter than C lightweight aggregate specimens by 13.5%.



(a)

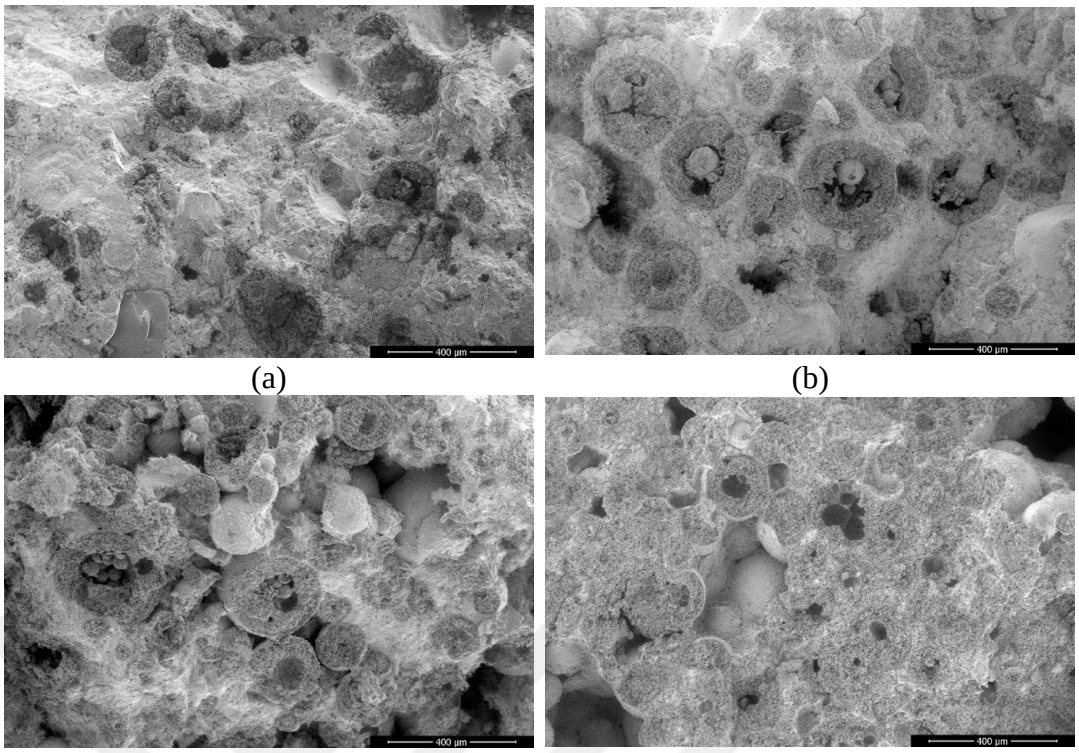


(b)



(c)

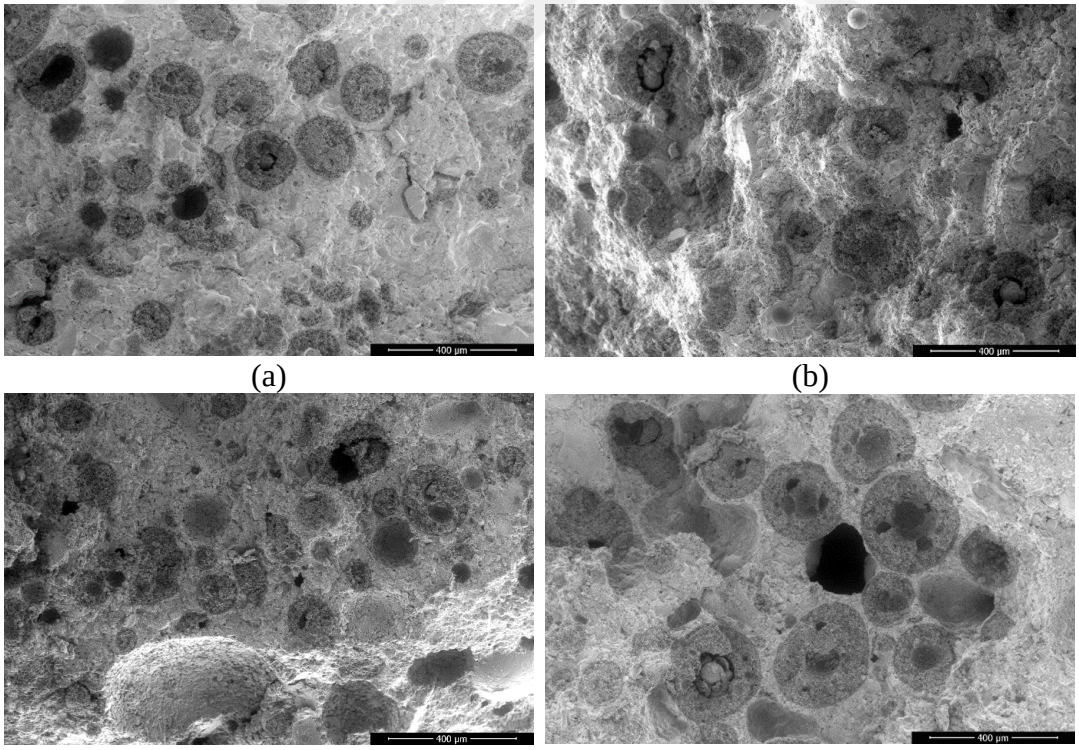
Figure 3.8 : Micro structure of PCM-free samples (a) C (b) FA (c) S.



(a) (b)

(c) (d)

Figure 3.9 : The micro structure of cement based aggregates (C-LWA) with the PCM amount a) 6% PCM b) 12.5% PCM c) 25% PCM d) 50% PCM.

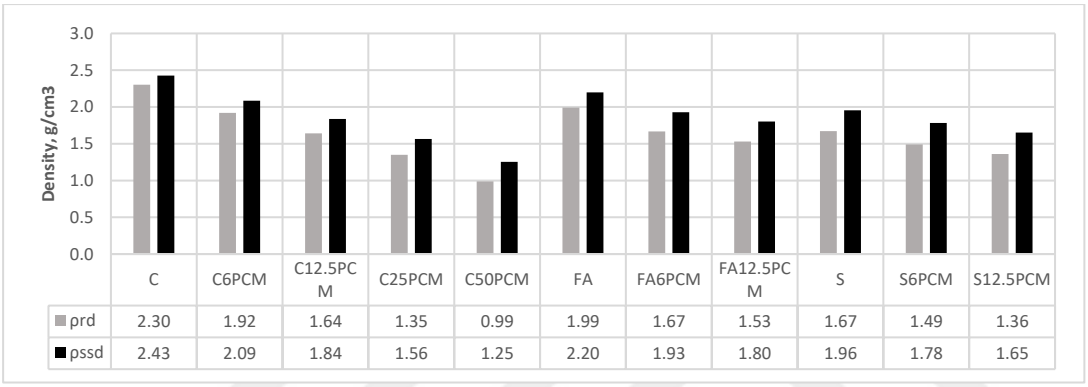


(a) (b)

(c) (d)

Figure 3.10 : The micro structure of cement and SCM based aggregates with the PCM amount (a) FA6PCM (b) FA12.5PCM (c) S6PCM (d) S12.5PCM.

PCM incorporation decreased the particle density gradually down to 0.99 g/cm^3. When three aggregate groups were examined among themselves, effect of PCM on density was observed the most significant at C-LWAs with a decline of 29% for the case of 12.5% PCM addition. A decline of 23% was observed in FA-LWAs, while this rate was nearly 19% for S-LWAs. In case of 6% PCM incorporation, C-LWAs and FA-LWAs showed similar values of decline on density around 16.5%. This decline has reached to 11% in the S-LWAs. When these values compared with densities observed in studies in literature [12], [13], [14], [39], it can be concluded that the incorporation of PCM makes it possible to manufacture lighter aggregates.



	C	C6PCM	C12.5PCM	C25PCM	C50PCM	FA	FA6PCM	FA12.5PCM	S	S6PCM	S12.5PCM
prd	2.30	1.92	1.64	1.35	0.99	1.99	1.67	1.53	1.67	1.49	1.36
pssd	2.43	2.09	1.84	1.56	1.25	2.20	1.93	1.80	1.96	1.78	1.65

Figure 3.11 : Particle density of the pelletized aggregates.

In Table 3.9, water absorption capacities of manufactured LWAs and porosity values are introduced. Since C-LWAs have a denser structure (Figure 3.8) than the other two groups (FA-LWA and S-LWA) on tested age, water absorption and porosity values were also lower. With the addition of 50% PCM, the water absorption capacity and porosity increased up to 5 and 2 times, respectively. On the other hand, in the S-LWAs, which have lower densities, the PCM addition did not create a significant increase in the water absorption capacity and porosity, although the absolute values were higher than C-LWAs and FA-LWAs. When 12.5% PCM was used, the increase in water absorption capacity and permeable porosity of S-LWAs was 27% and 3% respectively, while these increments were 71% and 32% for FA-LWAs and 118% and 56% for C-LWAs.

When the results are evaluated together, the different densities of cement, fly ash and slag may be addressed for the change of the physical properties of manufactured mixtures. Aforementioned water amount variation also explains the different

intergroup responses given to the PCM incorporation. The different micro structure properties obtained from the SEM analysis also support these results.

Table 3.9 : Water absorption capacity and permeable porosity of manufactured LWAs.

Aggregate type	Water absorption (%) WA	Permeable porosity (%) PP
C	5.5	12.6
C6PCM	8.6	16.6
C12.5PCM	12.0	19.7
C25PCM	15.8	21.4
C50PCM	26.9	26.6
FA	10.3	20.6
FA6PCM	15.6	26.0
FA12.5PCM	17.7	27.1
S	16.9	28.3
S6PCM	19.5	29.1
S12.5PCM	21.4	29.2

3.3.4 Mechanical properties of LWTA

Mechanical properties of LWTAs were determined from crushing strength tests conducted on manufactured aggregates. Test results given in Figure 3.12 are discussed below considering the effect of binder type, curing time and PCM ratio of the mixtures.

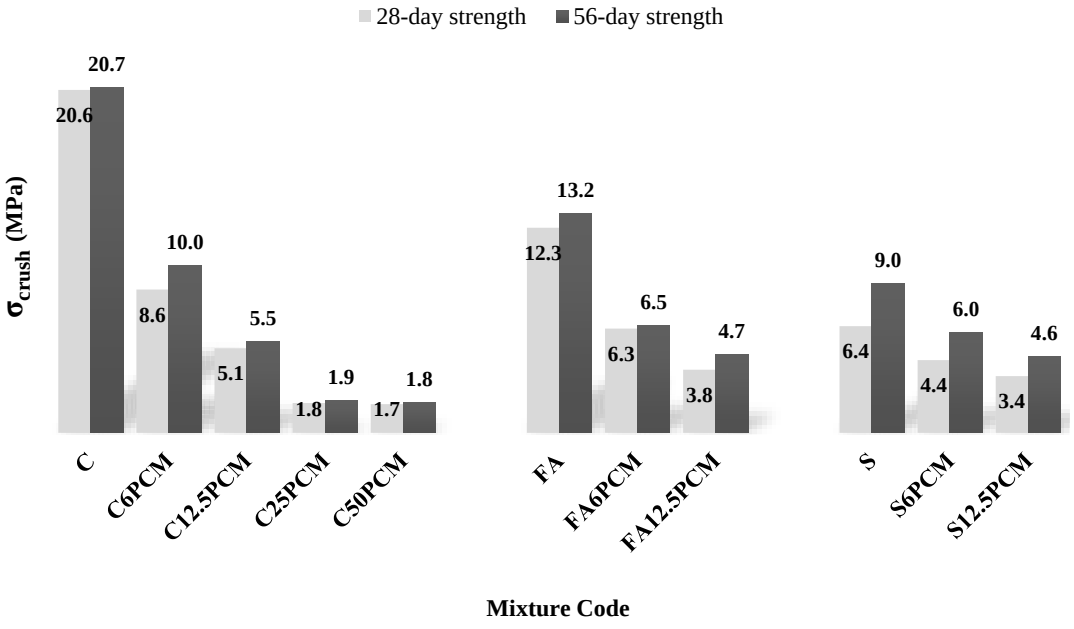


Figure 3.12 : Strength performance of the pellets at the end of 28 and 56 days.

3.3.4.1 Effect of binder type on the mechanical properties of aggregates

In this section, mechanical strength test results of the aggregates which were manufactured from the PCM-free mixtures are analyzed. When the results of the reference samples investigated, it is seen that pellets containing completely cement as binder exhibited the highest strength as expected. As seen in Figure 3.12, while the strength of the cement-bonded aggregates increased up to 20.7 MPa, the reference mixture of the group that contains slag together with cement exhibited only a 9 MPa strength at the end of 56 days. Utilization of slag as supplementary cementitious material caused 56% decrease in crushing value of the aggregates. When crushing strength values of aggregates containing fly ash are evaluated with reference to the cement group, a reduction of 36% in strength is obtained. Since the main idea in the aggregate pelletization is to recycle of the by-products such as fly ash and blast furnace slag, SCM amount in aggregates were kept relatively high in proportion to cement. These materials are known as pozzolans which are described in ASTM C125, as a siliceous or siliceous and aluminous materials that in itself possesses little or no cementitious value but will, in finely divided form and in the presence of water, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties [123]. Considering that the required calcium hydroxide is supplied from hydrating Portland cement's hydration, the limited amount of cement presented in the aggregate may be responsible from the relatively low strength of SCM based aggregates. Relatively high water/binder ratio of SCM based aggregates (Table 3.5) leading to a more porous structure (Table 3.9) (Figure 3.8) is another reason for strength drop (Figure 3.10). For a better performance, thermal treatment methods such as sintering, autoclaving and steam curing can be used but since the research involves PCM, these techniques with high temperatures are not preferred. When two mixtures were compared among themselves, it was found that the one that includes FA had 47% higher strength than the one includes GGBS. This strength difference between fly ash and slag based pellets can be explained by the difference of the water/binder ratio between the mixtures (Table 3.5).

3.3.4.2 Effect of curing time on the mechanical properties of aggregates

Experiments were carried out on 56-day samples in addition to the 28-day experiments, for a better understanding of the effect of fly ash and slag on the strength

at varying ages. At the end of the 56-day curing period which allows a better hydration of samples, all of the groups exhibited better strength results. The effect of the curing time reveals itself in the S-LWAs with a 2.6 MPa increase in strength which corresponds a gain of 40%. Although the ultimate strength of FA-LWAs and S-LWAs is close to each other, the fly ash based mixtures showed a better performance in early ages compared to the slag group. Reason of this early strength gain can be ascribed to the high Al_2O_3 and Fe_2O_3 content of the fly ash in comparison to the slag.

3.3.4.3 Effect of PCM ratio on the mechanical properties of aggregates

Mechanical properties of aggregates degraded regardless from binder type in case of PCM addition. Depending on the PCM ratio, the average 56-day crushing strengths varied between 1.8 MPa and 20.7 MPa for cement based mixtures, 4.7 MPa and 13.2 MPa for fly ash based mixtures and 4.6 MPa and 9 MPa for slag based mixtures. The strength loss of the manufactured aggregates reached up to 90% in the case of 50% PCM incorporation. In C-LWAs, crushing strength decreased by 52% with the addition of 6% PCM and strength loss increased gradually depending on the PCM percent of the mixture until 25% PCM. When PCM ratio was increased to 50% from 25%, crushing strength retained its value. Strength reduction of fly ash and slag based groups in the presence of 6% PCM was obtained as 51% and 33% relatively, while these ratios were approximately 64% and 49% in the case of 12.5% PCM incorporation. Although the strength loss of the FA-LWAs appears to be higher than that of the S-LWAs, Figure 3.12 shows that the PCM containing mixtures of both groups show approximately the same strength. This is because the reference mixture of FA-LWAs has a higher strength than the reference of S-LWAs. The relationship between the PCM rate of mixtures and the crushing strength are presented in Figure 3.13. The effect of PCM ratio on crushing strength of pellets was found to be more significant than effect of binder type. Strength decreased linearly with increase of PCM ratio. When PCM was used beyond 12.5%, a dramatic strength decrease was observed. The cause of this dramatic change can be attributed to disconnection of cementitious phase due to the interconnection of PCM particles with each other (Figure 3.9). This phenomenon is explained in more detail in the relevant section by microscope analysis results. As mentioned in section 3.3.1, with the increase of PCM amount, water amount that the mixture needs for pelletization increased (Table 3.5). Higher water / binder ratios that come out at high PCM ratios are another reason for the reduction in strength.

Different material content of mixtures resulted in different water needs and this makes it difficult to analyze the pure effect of the water / binder ratio on the results which was outside the scope of this study. So these two factors were evaluated together. PCM addition also reduced the binder amount of mixtures which is another cause of the decrease in strength. Systematic experimental studies can be planned as future work to evaluate the effect of binder and water amount itself to the mechanical properties of PCM integrated pelletized aggregates.

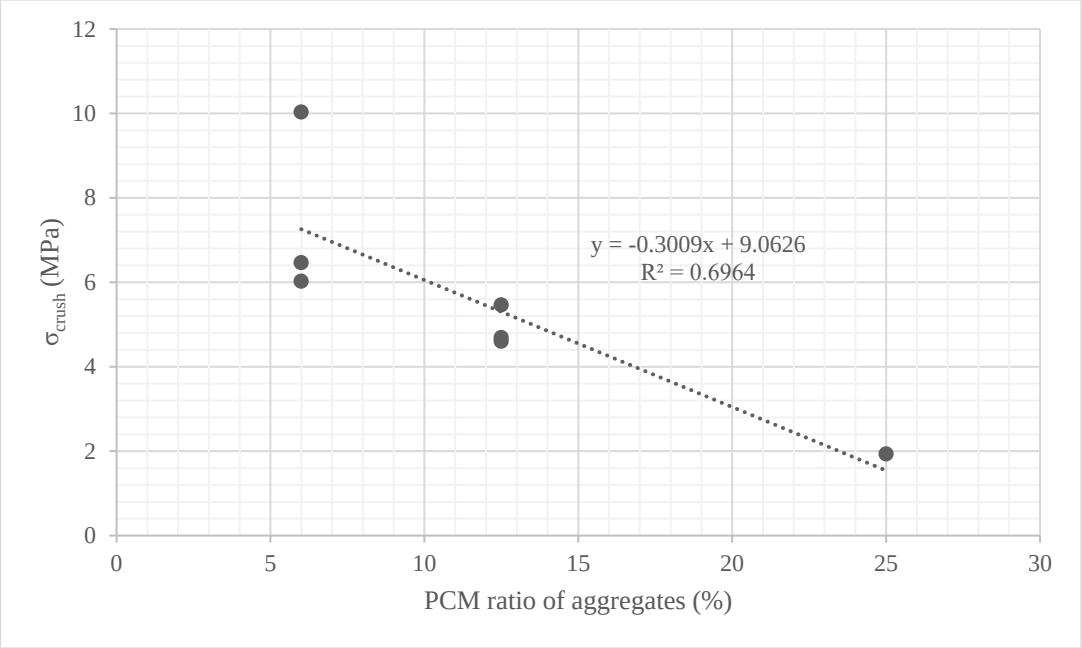


Figure 3.13 : Relation of 56-day crushing strength with PCM ratio.

In Figure 3.14, the linear trend between the strength of LWTAs at the 56th day and the oven-dried density of mixtures is presented. As seen from this figure, these two properties are highly correlated with each other. It can be concluded that, in general, as the particle density of the aggregates decreases based upon the increase of PCM ratio of the mixtures, the crushing strength of the aggregates is also decreases.

3.3.5 Thermal properties of LWTA

The thermal characterization of the LWTAs and the PCM used in the aggregates were both obtained by DSC analysis and the details of these results are presented in Table 3.10. In this table, information deduced from DSC curves including starting temperature of phase transition (T_{onset}), ending temperature of phase transition (T_{endset}), peak temperature of phase transition (T_{peak}) and phase change enthalpies (ΔH_{endoth} for melting process, ΔH_{exoth} for solidification process) is introduced. PCM used in the

study starts melting at 21.0℃ and completes the transition at 31.4℃ with a peak at 25.5℃. It starts to solidify at 22.8℃ and solidification ends at 10.7℃ with a peak temperature of 18.2℃. Measured melting and solidification enthalpies of PCM are 83.95 kj/kg and 91.32 kj/kg, respectively.

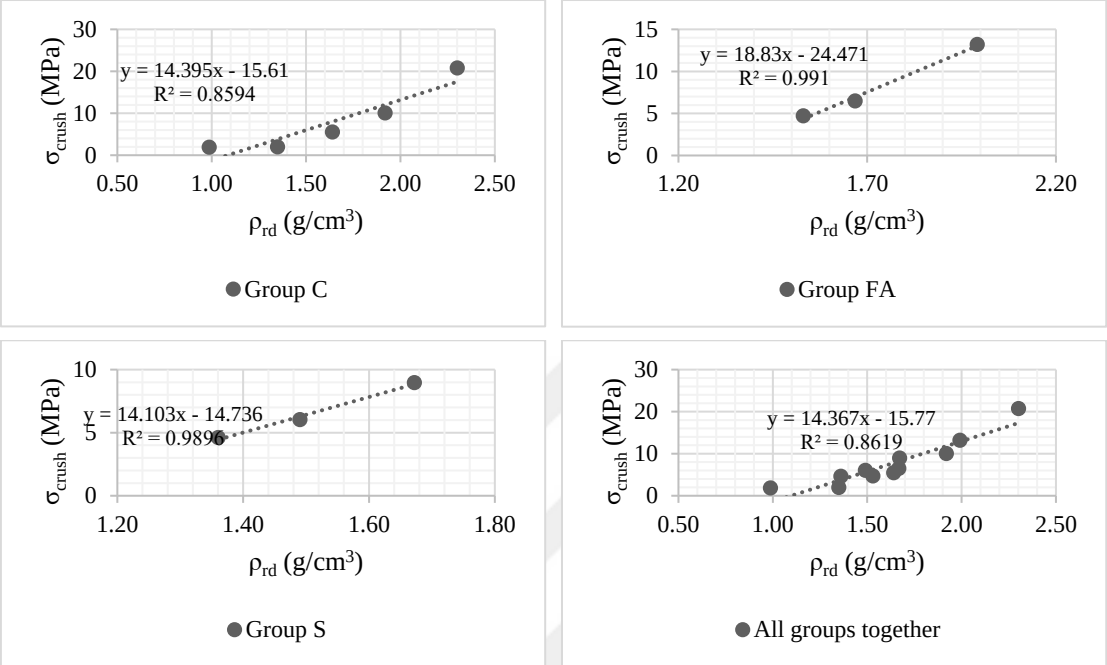


Figure 3.14 : Relation between the strength and the density of manufactured LWTAs.

Table 3.10 : Thermal properties of PCM used in the study and manufactured LWTAs.

Mixture Code	PCM Amount	Melting Process				Solidification Process			
		T_{peak} (℃)	T_{onset} (℃)	T_{endset} (℃)	ΔH_{endoth} (kJ/kg)	T_{peak} (℃)	T_{onset} (℃)	T_{endset} (℃)	ΔH_{exoth} (kJ/kg)
PCM	100	25.5	21.1	31.4	83.95	18.2	22.8	10.7	91.32
C	0	-	-	-	-	-	-	-	-
C6PCM	6	23.2	19.4	24.7	1.42	17.5	20.7	10.4	2.72
C12.5PCM	12.5	25.0	21.6	26.8	2.27	16.4	20.5	10.9	3.69
C25PCM	25	24.0	20.8	26.6	7.93	17.4	20.8	13.4	9.02
C50PCM	50	24.4	20.5	27.6	20.90	16.8	21.4	12.2	23.51
FA	0	-	-	-	-	-	-	-	-
FA6PCM	6	24.7	20.7	28.4	1.24	17.2	20.5	10.6	2.15
FA12.5PCM	12.5	24.8	21.3	26.4	2.64	16.7	20.7	13.3	3.11
S	0	-	-	-	-	-	-	-	-
S6PCM	6	24.1	19.6	25.7	1.59	17.1	20.0	10.5	2.31
S12.5PCM	12.5	23.1	20.0	24.9	2.56	18.3	20.9	14.3	2.66

The equivalent properties of each artificial aggregate group can be found in Table 3.10. When onset, endset and peak temperatures obtained from the analysis of the aggregate groups were examined together, similar results stand out in all groups. Average values of these temperatures for melting process are 20.5℃, 26.4℃ and 24.1℃ respectively. In the solidification process, onset, endset and peak temperatures of phase transition were obtained as 20.7℃, 12.0℃ and 17.2℃, respectively. When the relevant temperature range is considered, because the only phase changing material in the mixtures is the PCM, these results are consistent with the results obtained from pure PCM tests. The temperature interval of the phase transition during the melting process was found to be between 19.4℃ and 28.4℃. On the other hand, LWTAs completed the phase transition in between 21.4℃ and 10.4℃ when they cooled. The same super-cooling effect was also observed in the analysis of pure PCM (Table 3.10). This phenomenon can be attributed relatively higher values of the heating or cooling rate [88], [89]. Theoretical enthalpy values can be calculated by multiplying the mixture PCM ratio by the enthalpy value of pure PCM. When the experimental results (Table 3.10) are interpreted according to the theoretic values, a substantial decrease is seen. The reason of this may be the thermal conduction problems which may occurred as a result of the interaction between PCM particles and cementitious phase. Relatively higher values of the heating or cooling rates may also promote these problems as mentioned before. Besides, damaged PCM capsules both during the aggregate production process and during the sample preparation for the DSC experiments may affect the results negatively. Although the phase change enthalpies of LWTAs were lower than expected, they changed in accordance with PCM ratio of the mixture. Figure 3.15 shows the linear relation between enthalpy and PCM ratio of the mixtures. The average values of the melting and solidification enthalpies given in Table 3.10 were used in Figure 3.15. A high correlation was obtained between the PCM amount present in the aggregate and the thermal capacity of the aggregate. This shows that the PCM contributes the thermal performance of the LWTAs linearly with the usage ratio.

3.3.6 Physical properties of LACs produced using manufactured LWTAs

Physical properties of the concretes produced using manufactured LWTAs were presented in Table 3.11. The measured density values were in the range of 1.8-2.1 g/cm^3. Concretes having an oven-dried density of not more than 2.0 g/cm^3 and made with aggregates consisting entirely or partly of LWAs may be classified as lightweight

aggregate concrete (LAC) according to the EN 206 standard [124]. Concretes produced with manufactured LWTAs can obtain the LAC classification [except three specific LAC groups (C/C, C/C6PCM, C/FA)] since their densities are in the range of 1.8-2.1 g/cm^3 as presented in Table 3.10. 55% of the aggregate mixture used in concrete was composed of CEN standard reference sand. This sand has a higher density compared to the manufactured LWTAs, therefore it increased densities of concretes. Density values decreased proportionally with the density of used aggregate type (depending on its PCM content). Although no drastic change was observed in water absorption capacities as well as open porosities of the concretes, these values showed a slight increase in concretes produced with FA-LWA and S-LWAs.

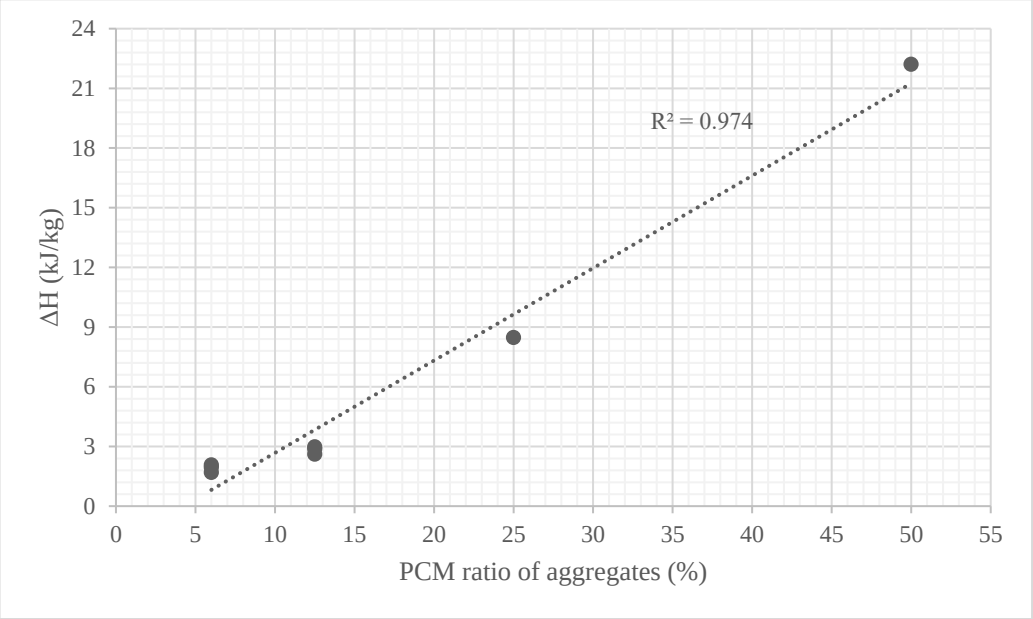


Figure 3.15 : Relation between the enthalpy and PCM ratio of LWTAs.

Table 3.11 : Physical properties of LACs produced using manufactured LWTAs.

Concrete name	Type of LWTA used	Oven-dried density (gr/cm^3)	Water saturated density (gr/cm^3)	Water Absorption (%)	Permeable porosity (%)
C/C	C	2.1	2.3	8.0	17.0
C/C6PCM	C6PCM	2.1	2.2	7.2	14.9
C/C12.5PCM	C12.5PCM	2.0	2.1	7.9	15.7
C/C25PCM	C25PCM	1.9	2.1	8.7	16.5
C/C50PCM	C50PCM	1.8	2.0	8.8	16.2
C/FA	FA	2.1	2.2	8.9	18.4
C/FA6PCM	FA6PCM	2.0	2.2	8.8	17.7
C/FA12.5PCM	FA12.5PCM	2.0	2.1	9.1	17.7
C/S	S	2.0	2.2	8.5	17.2
C/S6PCM	S6PCM	2.0	2.2	9.4	18.5
C/S12.5PCM	S12.5PCM	1.9	2.1	9.4	18.2

3.3.7 Mechanical properties of LACs produced using manufactured LWTAs

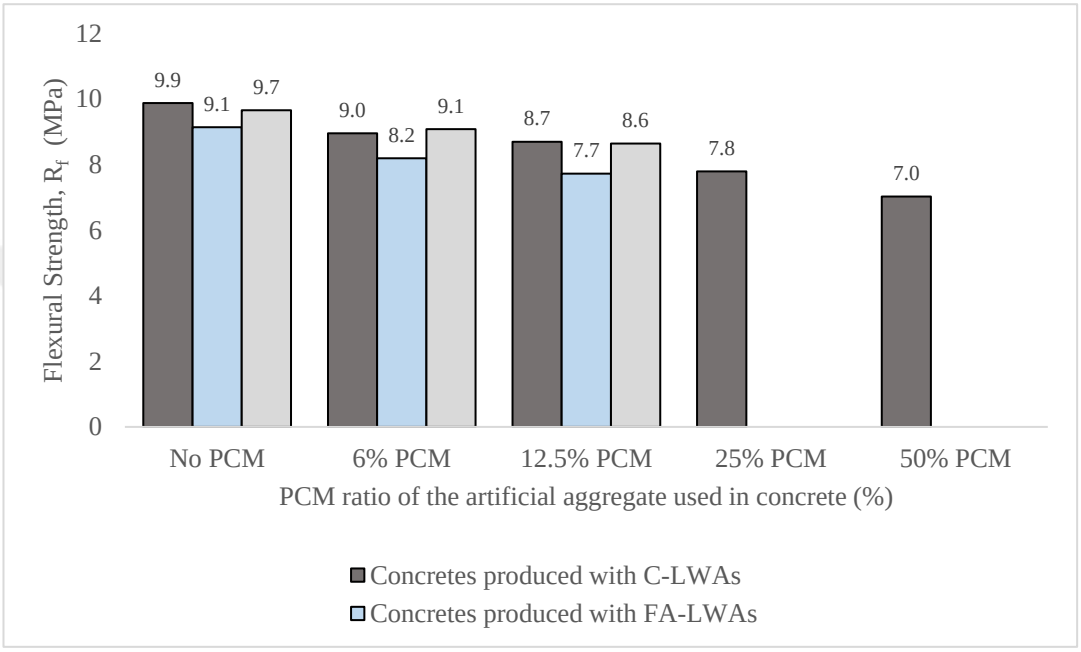
Mechanical tests conducted on the produced concretes reveal the decrease in the mechanical performances of the concrete with use of PCM. Results of the flexural and compressive tests performed on LACs are presented in Figure 3.16 (a) and Figure 3.16 (b), respectively. Values given in these figures are the average strengths of identical samples that belong the same concrete groups. Flexural strength values of the concretes were determined in between 7.0 MPa and 9.9 MPa. As seen from Figure 3.16 (a), concrete groups that were produced using C-LWAs and S-LWAs showed almost the same flexural performance. In any group, when PCM amount in the LWTA increased, flexural strength of the concrete decreased. Maximum strength drop was 29% in the samples produced using C50PCM. When the equivalent groups in terms of PCM ratio are compared among themselves, the highest decrease was observed in concretes produced with FA-LWAs and this decrease was 15% for concrete produced with FA12.5PCM aggregate.

The effect of the PCM ratio of LWTA to the average compressive strength of the concrete is presented in Figure 3.16 (b). Depending on the aggregate used, concrete compressive strengths varied between 33.9 MPa and 73.2 MPa for concretes with C-LWAs, 48.7 MPa and 65 MPa for concretes with FA-LWAs and 44.3 MPa and 64.6 MPa for concretes with S-LWAs. When PCM-free mixtures are compared among themselves, the strength values of concretes containing FA-LWA and S-LWA showed a decrease of about 12% compared to concretes containing C-LWA. Also PCM containing mixtures of these two groups showed approximate results. Therefore, this data shows that for all aggregate groups (C-LWAs, FA-LWAs and S-LWAs) as the PCM ratio of the aggregate in the concrete increases, both the flexural strength and compressive strength of the concrete mixture decreases. While this strength reduction was observed to be around 30% in the case of LWTA incorporating 12.5% PCM, it goes up to 54% in the presence of LWTA that contains 50% PCM.

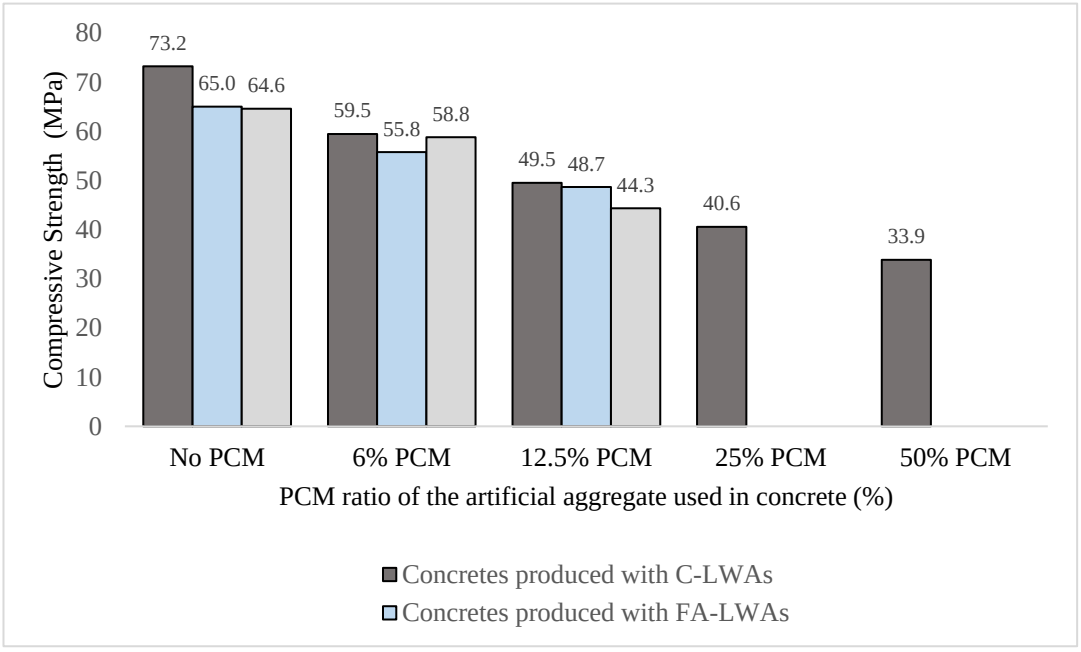
3.3.8 Microscopic analysis on LWTA concretes

The produced artificial lightweight aggregate can be identified as two phase material. First phase contains PCM, in addition to sand and binder while the second phase does not include PCM. The second phase is only utilized as shield to prevent the possible PCM leakage from the aggregate. Although microencapsulated PCM used in the scope

of this study, to create the possibility of using non microencapsulated PCM materials in artificial lightweight aggregate production, thin shield was created by using binder, sand SP and water. An example of PCM integrated lightweight aggregate cross section is given in Figure 3.17. Almost 0.5 mm thick shield was created in to prevent the possible leakage of the PCM materials from the lightweight aggregate. This also helps to increase the bonding between the PCM aggregate and cement paste.



(a)



(b)

Figure 3.16 : Mechanical performances of the concretes produced with manufactured LWTAs on 28th day (a) Flexural (b) Compressive.

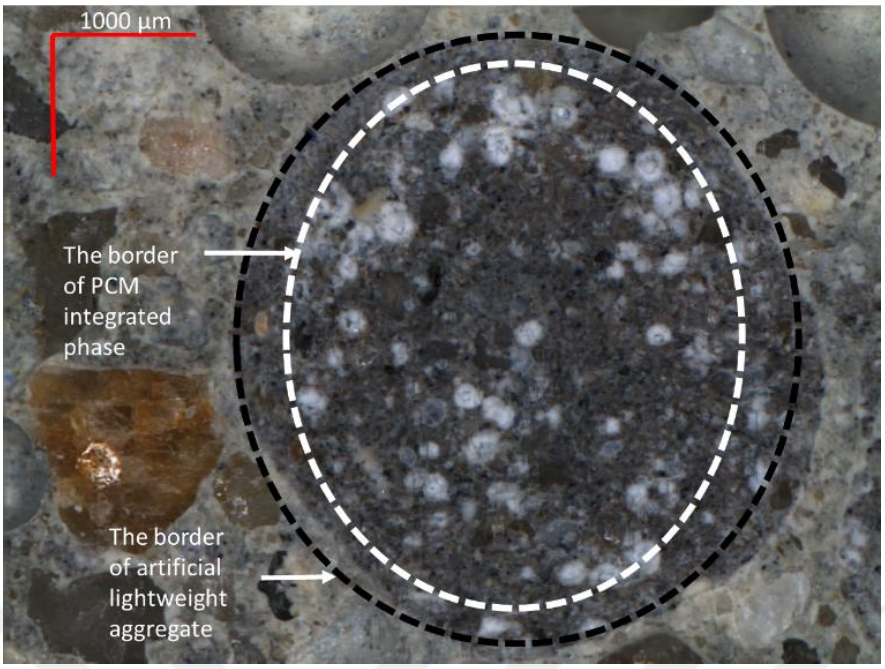


Figure 3.17 : The cross section of PCM integrated lightweight aggregate.

The strength deduction in aggregate samples were much significant than concrete samples when PCM is used. This difference is arisen from the different failure mechanisms between aggregate and concrete strength tests. In crushing strength test of aggregate, failure is occurred due to the tensile stresses whereas the compressive stresses are responsible from the failure of concrete samples. Another reason of the prominent strength drop in the aggregates is the interface failure between PCM and matrix phase of the aggregate. Cross sections of the concretes under stereo microscope is shown in Figure 3.18. Left column shows the concretes produced with control samples which do not include PCM. Thermal cycling allowing phase change of PCM, due to the natural thermal changes to which the aggregates are going to be exposed during their lifetime did not affect negatively the interface between aggregate and cement paste.

Micrograph of cement binder based lightweight aggregates is shown in Figure 3.19. It is obvious that the distribution of PCM particles are well in the aggregates including 6% and 12.5% PCM. PCM particles are very close to each other at usage of 12.5% and over. As explained above, SEM analyzes have shown in detail the convergence of PCM particles encountered at high utilization rates (Figure 3.10) This structure of PCM particles disconnects the continuous load carrying cementitious matrix. Disconnection of cementitious phase results as decrease in the strength of the

aggregates. The dramatic decrease in strength values at 50% and 25% PCM materials can be explained with the distance between PCM particles.

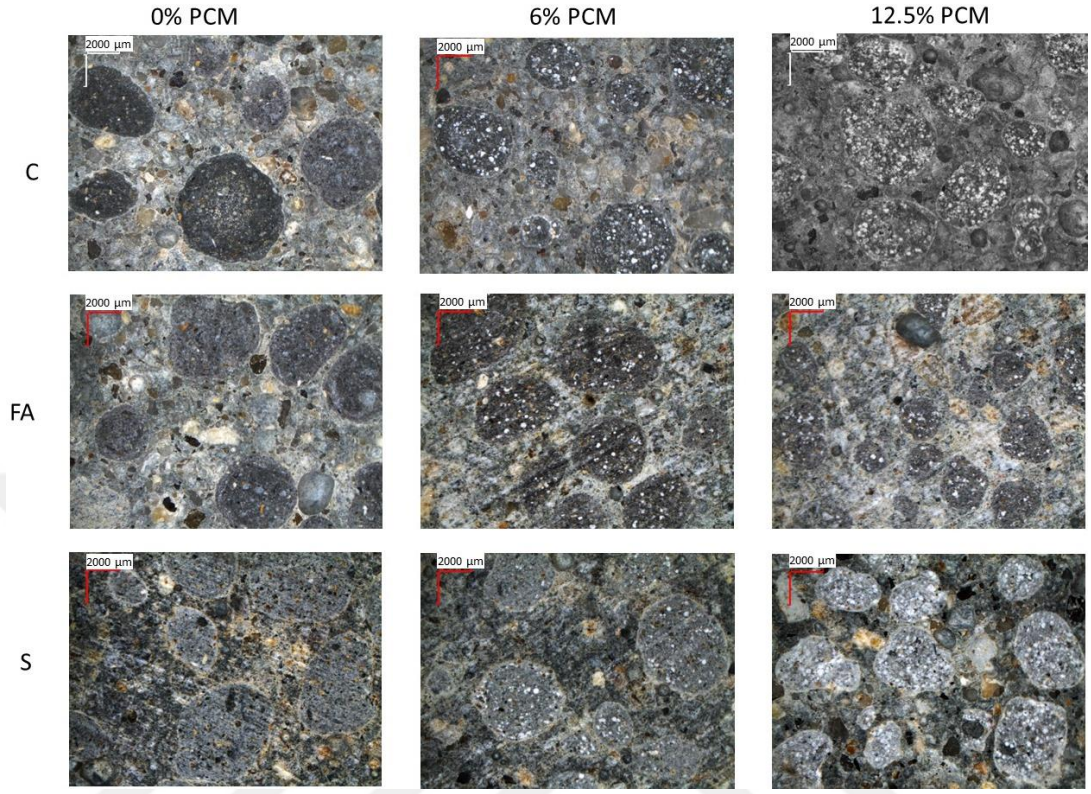


Figure 3.18 : Concrete cross sections containing artificial lightweight aggregate.

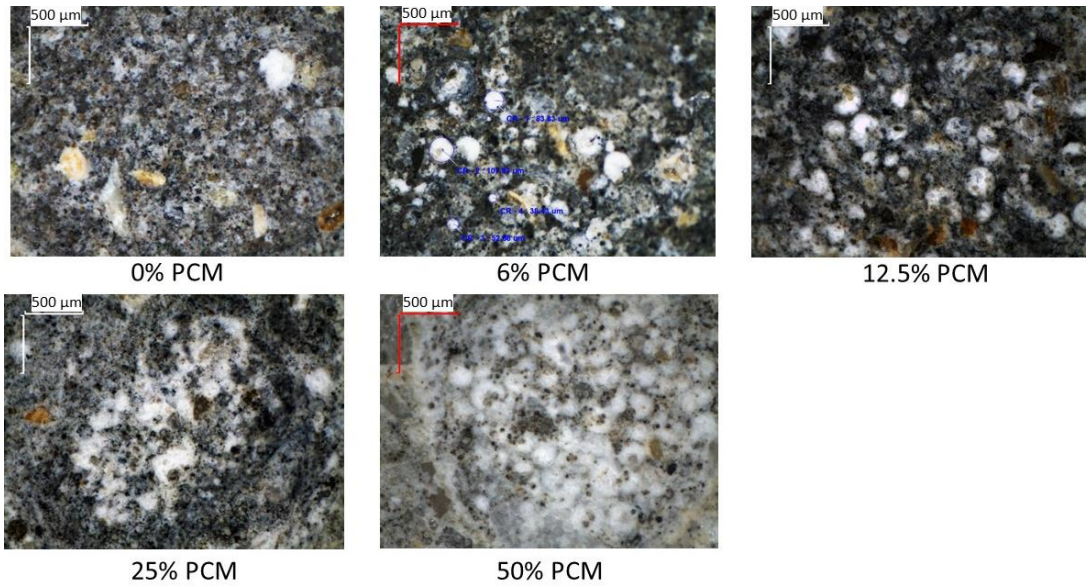


Figure 3.19 : Micrograph of cement binder based lightweight aggregates.

In higher usage ratios, the circular shape of PCM particles have been destroyed. Microencapsulated PCM particles touch to each other which may create additional stresses on the microencapsulation capsule [Figure 3.9 (d)]. Additional stresses over

the capsule can result as detoration of capsule and leakage of PCM through cement paste phase.

Micrograph of cement and SCM based light weight aggregates are shown in Figure 3.20. Since production of 25% and 50% PCM aggregates were not possible, only 0%, 6% and 12.5% PCM aggregates were investigated for SCM binder aggregates. The distribution of the particles in SCM based lightweight aggregates similar to cement based ones. Until 12.5% usage the distribution was homogenous.

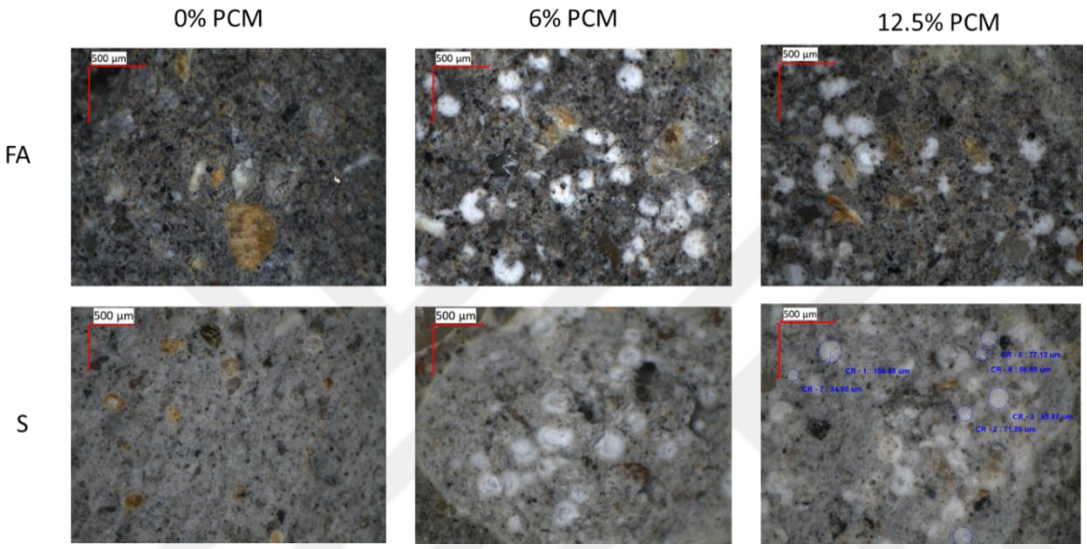


Figure 3.20 : Micrograph of cement and SCM based lightweight aggregates.

3.4 Conclusions

In this study, we produced cold bonded aggregates utilizing SCM and PCM. Physical, mechanical and thermal properties of LWTAs were studied systematically. Performance of the produced LWTAs as a concrete component were determined through concrete production and testing. Within the limits of this experimental study, the following conclusions can be drawn:

- PCM reduced the pelletization tendency while duration of the production process increased. It also affected to the shape of grains. Pellets became smaller and the shape of the particles started to flatten with the increase of PCM ratio in the mixture. This inconvenience was attributed to the hydrophobic nature of the PCM.
- Moisture level required for pelletization changed along with the components of the mixture. This level was highest for the slag based mixtures. Fly ash

aggregates also had a higher water need compared to the mixtures which only contains cement binder. PCM incorporation augmented the water demand regardless of the binder type.

- PCM utilization decreased the density down to 0.99 g/cm^3. Compared to equivalent mixtures, the lowest density values were measured in slag-based aggregates. Water absorption capacity and open porosity of the slag-based aggregates were also higher than the other groups. It can be concluded that the use of PCM together with by-products such as blast furnace slag or fly ash, has been instrumental in achieving light artificial aggregates.

- Cement based aggregates showed the highest strength as expected. Depending on PCM ratio, crushing strengths varied between 1.7 MPa and 20.7 MPa. Increase of PCM ratio resulted in a gradual decrease in crushing strength of the LWTA. When PCM was used beyond 12.5%, PCM particles were clustered very close to each other and lose their spherical structure. This disrupted the continuous structure of the load carrying cementitious phase and caused a dramatic decrease in strength of LWTAs. In the PCM-free series, the fly ash mixture showed higher strength results than the slag mixtures, while the performance of the two groups were closer to each other in the case of PCM incorporation. Another important point was the effect of curing time on slag-based aggregates which leads a 40% increase in strength. When the strength results were evaluated together with the physical properties of the aggregates, a high correlation was found between the strength and the density of the aggregate particles.

- PCM incorporation increased the latent heat capacity of the LWTAs. Although the increase was systematic, the observed enthalpies remained below the theoretical values. The reason of this may be the interaction between PCM particles and cementitious phase. In addition, inconveniences encountered in production and sample preparation stages also affected the results. This issue should be investigated in detail in further studies. Considering that the generated aggregates are not intended to be used for direct energy storage, it can be concluded that these LWTAs will contribute to thermal comfort by blocking excess solar energy from outside the building, within the concept of passive construction.

- Cold bonded lightweight PCM aggregates produced within this study were used to produce LAC and it was observed that the density of LAC decreased with the density of used aggregate type depending on its PCM content. Water absorption capacity and permeable porosity of concretes did not show a significant change.
- Mechanical performance of the LACs decreased depending on the PCM ratio of the used LWTA. The maximum reduction, which is 29% for flexural strength and 54% for compressive strength, was observed in concretes produced with LWTA having a PCM ratio of 50%. Although the PCM causes a significant decrease in compressive strength, the minimum strength value obtained as 33.9 MPa in this study, which is a sufficient strength for many structural applications.

Within the limits of the ratios used in this study, the maximum amount of microencapsulated PCM to be used in cold bonded LWTA can be stated as 12.5%. Greater amounts not only cause non-uniform, improper products, but also reduce mechanical properties. Since the aggregate becomes very weak when PCM is used above the specified amount, the failure of concrete occurs through the aggregate.

4. PERFORMANCE OF GLASS FİBER REINFORCED CEMENT COMPOSITES CONTAINING PHASE CHANGE MATERIALS[1]

Abstract

The use of solar energy has become a promising method to reach energy efficient solutions throughout the last century. Improving the thermal properties of building members will contribute to the efficient use of energy by preventing heat gain/loss through the building envelope. Phase change materials (PCMs) are favorable materials for thermal applications because of their large contribution to the thermal mass of a building and thus providing "inertia" against temperature fluctuations. In this experimental study, glass fiber-reinforced cementitious composites were modified using a commercial microencapsulated PCM to improve their thermal performance. The thermal and the mechanical performance of the composites were investigated. With the use of PCMs, the latent heat capacity of the composites increased while the thermal conductivity decreased. Conversely, the mechanical properties of composites declined with increases in PCM amount. The main conclusions of the experimental study are as follows: PCMs provide a great potential to fiber reinforced cementitious composites to support the efficient use of energy, thereby enhancing the thermal features of the composites. Performance of PCM composites shows a serious variation according to different service temperatures. Therefore, effect of service temperature must be considered particularly with respect to safe utilization of these materials.

Keywords: PCM, fiber-reinforced cementitious composites, latent heat capacity, energy efficiency

[1] This chapter is based on the paper "Tuncel, E.Y., Pekmezci, B. Y. (2019). Performance of Glass Fiber-Reinforced Cement Composites Containing Phase Change Materials. *Environmental Progress & Sustainable Energy, 38*. doi:10.1002/ep.13061"

4.1 Introduction

The utilization of fibers in building materials has evolved into a highly practical and well-recognized reinforcement method to improve certain properties, especially in brittle mediums. Fibers improve the post-elastic properties of the matrices in which they are embedded [125], [126], [127], [128]. After the invention of alkali- resistant (AR) glass fibers, they became a common reinforcement material for both precast and in-situ applications such as external claddings, facade plates and other applications including electrical utility products, e.g., trench systems and distribution boxes, as well as in surface bonding and floating dock applications [128], [129], [130], [131].

Glass fiber-reinforced cement composites (GRCs) are very popular materials, especially in cladding applications because of their excellent mechanical properties, manageable consistency, and high fire and corrosion resistance. In the modern construction industry, buildings elements are designed not only as structural members but also as building envelope elements that protect the property from noise, temperature variations and weather disturbances, such as rain and snow [132]. Designing the cladding elements with additional features that improve their thermal or acoustic performance may offer smart solutions in the way of contribution to the comfort and budget of residents by providing efficient use of energy.

Energy is a crucial topic because it effects the economic and social development of the countries and because it is essential for a high-quality human life. It is a key parameter for all consumptions and production processes contributing to growth and human activities [41], [42]. Unsustainable consumption is a major cause of global environmental deterioration that leads to overexploitation of renewable resources, and fossil fuel-based pollution [133].

Buildings consume 32% of the global final energy demand and are responsible for 30% of the energy-related CO_2 emissions. In addition, approximately two-thirds of halocarbons and approximately 25%–33% of soot emissions come from building-based activities. Furthermore, the building sector is responsible for 23% of the global primary energy consumption and 30% of the global electricity consumption [134]. These data show that the control of the building-based energy quality and quantity is necessary for a clean and developed World. By improving the energy efficiency of

structures, the total energy efficiency of a society can be increased and the economic burden attributed to the building sector may be reduced [135].

Use of solar energy has become a promising method to reach energy efficiency goals throughout the last century. To take direct advantage of sunlight, the use of the solar panel systems in buildings has been a very classical method for many years [136], [137]. Passive buildings that offers the planar surface of the entire structure is another way to utilize solar power. Structural elements such as walls, windows and roofing are designed to collect sunlight during the day; store the energy in their body and return it to the building environment when the temperature drops below a certain level in the passive buildings. On hot summer days, they protect the interior from excessive solar heat [15] and they protect the interior from excessive cold during the winter. In prior centuries, structural elements made out of the traditional building materials, such as stone, marble and concrete, maintained the thermal comfort of buildings by their high heat capacity resulting from their large and bulky nature [138]. With the lighter structural envelopes offered by modern construction techniques, insulating materials and heating systems inside buildings have become preferred because of their lower production cost and faster construction schedule. Energy expenditures became important because of financial and environmental pollution concerns. To decrease the energy expenditure of modern buildings, phase change materials (PCMs) have been investigated for use in energy efficient buildings. PCMs have a high latent heat storage capacity. This latent heat storage can be expressed as the heat absorbed or released when the PCM changes its phase from a solid to a liquid (or a liquid to a gas) or vice versa at a constant (approximately) temperature. These materials have been used to collect excessive solar heat that enters the room during the day. With the ability to store solar energy during the day, PCMs minimize excessive indoor heating. At night, by releasing the stored energy to the building environment, they will prevent the indoor temperature from falling to an inconvenient level. By this cyclic behavior, PCMs help regulate the inside temperature within a desirable comfort zone [138], [139]. PCMs can be characterized by their transition temperature range, their transition related enthalpies (a measure of the internal energy storage), and their thermal conductivity that is related to the energy transfer rate. These features provide the ability to reduce the temperature peaks and valleys inside buildings, leading to less temperature variability, greater indoor comfort and major energy savings [107].

There are several methods reported in the literature to utilize PCMs in structures to regulate temperature changes of the indoor environment [140], [141]. PCMs can be used directly as a component of wallboards, floor tiles, mortars or concretes [24], [88], [142]. In addition, impregnation or immersion techniques can be used to advantage with PCMs [18]. In the case of direct use, the transition of a PCM from the solid to the liquid phase may cause some leakage problems in the medium. To use PCMs directly in mixtures such as mortars or concrete, some precautions must be taken to ensure a proper and long service life. Encapsulation is one of the methods to prevent PCM leakage. A permanent capsule surrounds the PCM core, preventing the PCM from dispersing. In addition to leakage protection, capsules provide the space required for PCM volume changes that occur during the phase transformation process. Unfortunately, the extra voids created by microcapsules negatively effects the thermal conductivity and mechanical performance of the system [29], [107].

There are many experimental studies in the literature focused on the thermal properties of PCM-containing building materials [46]. Memon et al. [92] studied the thermal performance of a lightweight aggregate concreate containing macro encapsulated paraffin – aggregate through small scale experiments. They reported a 4.7 ℃ decrease in the interior temperature at the room center. They also studied the economic and environmental aspects of the proposed PCM application and the application was proven to be feasible in Hong Kong. Hunger [88] et al. produced self-compacting concretes (SCCs) containing microencapsulated PCM. According to their work, the use of PCM did not affect the fresh concrete properties of the SCC but increasing PCM dosages lead to significantly lower compressive strengths. A 13% decrease for each additional percentage of PCM were reported. On the other hand, the specific heat capacity of the mixtures increased with the increase of PCM (up to 3.5 times for the 5% PCM content) and thermal conductivity of the mixtures reduced by increasing PCM dosage. Meshgin and Xi [143] also used PCM in Portland cement concrete both as a replacement of a certain percentage of fine aggregate (sand) and as an additive in the concrete mixture in this research. They have also reached similar conclusions about the general effects of PCM on mechanical and thermal behavior of concrete and reported that the strength reductions associated with the PCM replacement method are less than those of the PCM additive method. Zhang et al. [66] conducted a research about the thermal energy storage cement mortar (TESCM) fabricated by integrating

ordinary cement mortar with a composite PCM based on n-octadecane and expanded graphite (EG). Increase of the mass percentage of PCM composite led a decrease in compressive strength, apparent density, thermal conductivity and indoor peak temperatures of the test rooms. Cunha et al. [144] tested the thermal and mechanical performance of mortars made with PCM and different binders. They also conducted cost analysis based on energy consumption of mortars and energy price in Portugal. They concluded that the incorporation of the PCM microcapsules in mortars leads to a decrease in the energy consumption cost in the spring, autumn and summer, due to the decrease of heating and cooling needs, and consequently decrease of HVAC systems operation. Solgi et al. [96] also reported serious reduction values up to 34% in annual energy consumption of Iran, related to the use of PCM.

Although there is a common conclusion that PCMs adversely affect the mechanical properties, the effects on fiber-reinforced cementitious composites were not recorded [66], [88], [143]. This experimental work investigated the thermal and mechanical properties of GRCs, which are recognized for their enhanced mechanical strength and energy absorption capacity, in the presence of microencapsulated PCM over different service temperatures.

4.2 Materials and Methods

This section presents the experimental work conducted to understand the effects of microencapsulated PCM on the performance of GRCs. The objective is to determine the contribution of PCM to the thermal performance of the composites, as well as to the mechanical properties that are vital for structural materials. GRCs were modified using a commercial microencapsulated PCM to improve their thermal performance. The thermal properties of these mixtures were characterized using differential scanning calorimetry (DSC) method and conductivity analysis. Small scale cubicles were constructed and the temperatures inside the cubicles and the outside environment were monitored throughout natural daily cycles. The mechanical properties of the composites were also determined to evaluate the effects of PCM additions to GRCs. The effects of the PCMs incorporation on the compressive and flexural strengths, specific fracture energy and strain capacity were evaluated.

4.2.1 Material properties

In the manufacturing of the composites, CEM-FIL Anti-Crak HP 74/12 chopped glass strands were used as the reinforcement. For a better bending performance, chopped glass fibers were opted in the study. The matrix was produced using CEM I 42.5 R type Portland cement binder with a specific gravity of 3.14 g/cm^3 and natural quartz sand with a specific gravity of 2.65 g/cm^3. The manufacturer-supplied properties of the fibers are presented in Table 4.1. Table 4.2 shows the grain size distribution of natural sand. To address workability concerns, a polycarboxylate-based water reducer (WR) admixture was also used. Micronal DS 5008 powder-type microencapsulated PCM was utilized to enhance the thermal performance of the panels. Microencapsulated PCM is preferred in that it is easily accessible and practical for use with cementitious composites. Figure 4.1 provides a photograph of the PCM used in the mixtures.

The properties of PCM (provided by the manufacturer) are presented in Table 4.3 [145].

Table 4.1 : Properties of glass fiber.

Fiber Length	12 mm
Aspect ratio (length / diameter)	74
Filament Diameter	14 - 19 μm
Loss on Ignition (%) (ISO 1887 : 1995)	0.80 - 2.00
Moisture (%) (ISO 3344 : 1997)	0.50 max
Specific Gravity	2.68 g/cm^3
Material	Alkali Resistant Glass
Chemical Resistance	Very high
Modulus of elasticity	72 GPa
Tensile Strength	1700 MPa

Table 4.2 : Grain size distribution of the natural sand used in the matrix mixture.

Material	Percent Finer, %						
	Sieve Size, mm						
	16	8	4	2	1	0.5	0.25
Natural Sand	100	100	100	99	99	98	58

Table 4.3 : Properties of PCM.

Product type	Powder
Melting point (approx. in °C)	23
Overall storage capacity (approx. in kJ/kg)	135
Latent heat capacity (approx. in kJ/kg)	100

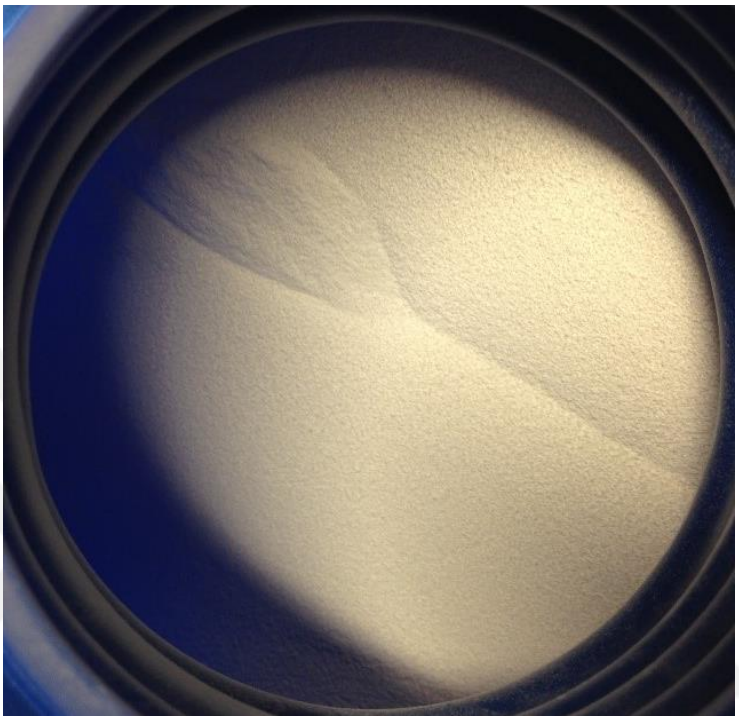


Figure 4.1 : Microencapsulated PCM used in mixture.

4.2.2 Design and preparation of composite panels

While designing composites, an ordinary glass fiber reinforced cement composite design which is already used in the industry for an improved bending performance was used. This design has been tried to be enhanced in terms of thermal properties using PCM in various quantities. In design of the composite panels, two different PCM amount were used (4% and 8% of total mass). Reason of limiting the PCM amount with 8% was due to the increase in water requirement of the mixtures in the higher PCM ratios. A plain mixture that did not include PCM was also produced for comparison. In all of the mixtures, the fiber ratio was maintained at 2% of the total volume. This fiber ratio was chosen in terms of the ability of the glass fibers used to be effective in the bending performance of the composites. The water/cement ratio was 0.40 and the consistency of the mixtures was adjusted using a WR admixture. The mixture containing 8% PCM, required additional water to achieve acceptable workability. In the study, the fact that the ratio of fiber was determined by the total

volume while the PCM ratio was determined by the total mass made the amount of mixing water insufficient in terms of workability in case of using 8% of PCM. This case can be explained by the extra volume introduced by the PCM which has relatively low density comparing to other materials in the mixture. This incremental water lowered the cement and sand amount in the actual mixture. Details of the mixture proportions are provided in Table 4.4. All of the mixes were prepared at 30°C using a 10-liter capacity laboratory mixer with vertical rotation axis by forced mixing. The workability was kept constant at 14 mm [146]. To prevent the damaging effect of the mixer paddles on the glass fiber filaments, hand mixing was utilized after the addition of the fibers. A photograph of the fresh mixture produced in the laboratory can be observed in Figure 4.2.

Table 4.4 : Mixture proportions.

Mixture #	Cement kg/m3	Sand kg/m3	water/cement ratio	PCM (% total w.)	Glass fiber (% total vol.)	WR admixture (% cement w.)
I	666	1332	0.4	-	2	2
II	608	1216	0.4	4	2	2
III	560	1120	0.5	8	2	2

After mixing, the fresh mixtures were cast into 350x350x20 mm plates and 160x40x40 mm prisms. The plates were intended to be used to construct cubicles for the temperature monitoring and as flexural test specimens. The prisms were produced for the compressive tests. Specimens were cured in saturated lime water at a constant temperature of 23±2 °C until tested. One day prior to flexural testing, the plates were cut using a diamond saw into 350x45x20 mm prisms. The 160x40x40 mm prisms were also cut into 40 mm cubes for compressive tests.

Figure 4.2 : Fresh mixture produced in laboratory mixer.

4.2.3 Methodology

The thermal properties of the PCM used in this research and the fiber reinforced PCM composites were analyzed by Differential Scanning Calorimetry (DSC). A reference composite containing no PCM was also tested in addition to those containing the PCM. DSC test samples were prepared by crushing the composite specimens used in the mechanical tests into small particles. DSC tests were conducted using a Shimadzu DSC-60A Differential Scanning Calorimeter under a nitrogen atmosphere. Temperature range of the calorimeter is -140 to 600°C and its calorimetric measurement range is ±150 mW. The DSC sample mass was approximately 30 mg. The sealed sample (placed in an aluminum pan) was placed in the calorimeter oven together with an empty reference pan. The selected temperature range to study the thermal transitions of the PCM was -10°C to 40°C. The dynamic scan was started by cooling from 40°C to -10°C and continued with dynamic heating segments that had heating or cooling rates of 5°C/min. The test procedure was repeated three times for every composite type, with virgin samples each time. The phase change temperatures and solidification-melting enthalpies were obtained from the heat flow measured during the tests. DSC test setup is shown in Figure 4.3.

Figure 4.3 : DSC test setup.

The thermal conductivity tests were conducted according to ISO 8301:1991 using a heat flow meter apparatus (Ahlborn) on plain and PCM composites [147]. Specimens

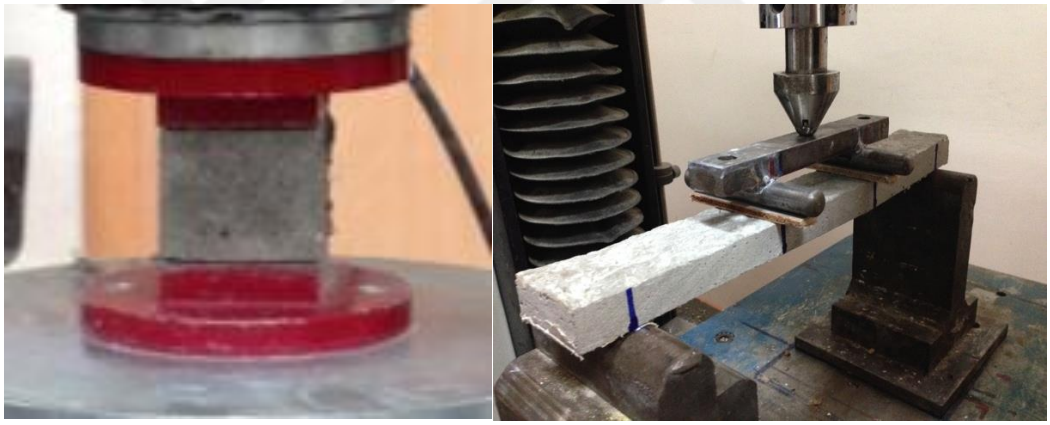
with dimensions of 350 x 350 x 25 mm were cast for the thermal conductivity measurements and were kept in saturated lime water at 21±1 °C temperature for 28 days. Specimens were dried at 60°C until they achieved constant weight to avoid any moisture-based test error. Testing began when the specimen came to room temperature. Determination of the thermal conductivity was performed at a steady state condition with a mean temperature of +2.5 °C at the center of the cross-section of sample and a measured temperature gradient of 5 °C.

To simulate the effect of PCM on the thermal performance of a building, cubicles having 310 mm inner dimensions were prepared for temperature monitoring using the plates described in section 4.2.2. The exterior dimensions of the cubicle specimens were 350 mm. Three cubicles representing each composite type were built using the panels prepared in the laboratory. Cubicles were sealed using silicone. The free-floating temperature inside the cubicles was monitored over a period of 60 days during the summer session. Cubicles (shown in Figure 4.4) were placed on the roof of the laboratory during monitoring to prevent a shading problem. Thermocouples and a Testo 177-T4 data logger which has 4 channels with 4 probe inputs and a memory for up to 48,000 readings were utilized to monitor the temperature inside the cubicles during the natural day and night cycles. Thermocouple which monitors the free-floating temperature inside the cubicle is fixed to the center of the cubicle so that the measuring tip is in contact with the air only. The temperature of the outside environment was also monitored.

Figure 4.4 : Cubicles for temperature monitoring.

The mechanical properties of the composites were based on the 28-day uniaxial compressive strength test and the simple beam four-point bending test. Compressive

strength tests were conducted on 40 mm cube specimens. For flexural tests, beam specimens of 45 mm width and 20 mm height were used. The length of the beam was 350 mm while the span length was 300 mm. A closed-loop testing machine (INSTRON 5500R) which has a 100 kN loading capacity was used in both tests and load-deflection curves were generated in the flexural tests. The specific fracture energy values (W_f) of the specimens were also determined in accordance with the recommendation of the RILEM 50-FMC Technical Committee [148]. Photographs of both compressive and flexural test setups are introduced in Figure 4.5 (a) and Figure 4.5 (b), respectively. Tests were conducted on two identical samples at varying temperatures, to observe the mechanical behavior of the composites at different temperatures that were above and below the melting temperature of the PCM (approximately 23°C). Five individual specimens were tested for each group. Specimens were conditioned for 24 h at 50°C and 10°C prior to testing. The results from different temperatures were compared to evaluate the temperature performance variation of the mixtures.



(a) (b)

Figure 4.5 : (a) Compressive test setup (b) Bending test setup.

4.3 Results and Discussion

4.3.1 Thermal experiments

4.3.1.1 DSC analysis of PCM

The thermal characterization of the PCM used in the composites was obtained by DSC analysis and the solidification and melting curves are presented in Figure 4.6. The details of these results including the onset temperature (T_{onset}), endset temperature (T_{endset}) peak temperature of phase change (T_{peak}) and latent heat (ΔH) for both the melting and solidification processes that were obtained from the DSC curves shown in

Figure 4.6 are presented in Table 4.5. The upper curve in Figure 4.6 shows the PCM solidification process. The PCM starts to solidify at 22.93°C and completes its phase change at 10.41°C, with a peak temperature of freezing at 18.22°C. The solidification enthalpy for this temperature range that was quantitatively estimated by using a linear approximation for the base line below the solidification peak was calculated to be 98.20 kJ/kg. In melting process (the lower curve in Figure 4.6), the phase change occurred between 20.45°C and 31.31°C with a peak temperature of 25.54°C and a melting enthalpy ΔH_{endoth} for this temperature range that was estimated to be 90.79 kj/kg. The difference between the melting and solidification peaks can be attributed to the selected heating or cooling rate. Hunger et al. [88] stated that higher values of the heating or cooling rate lead to broader melting ranges and that super-cooling effects can be observed because of the heating or cooling rate. This behavior is normally not expected when a lower heating or cooling rate of 0.1 to 0.5°C/min is applied. The enthalpy value that was observed is close to the value that was declared by the manufacturer (100 kj/kg).

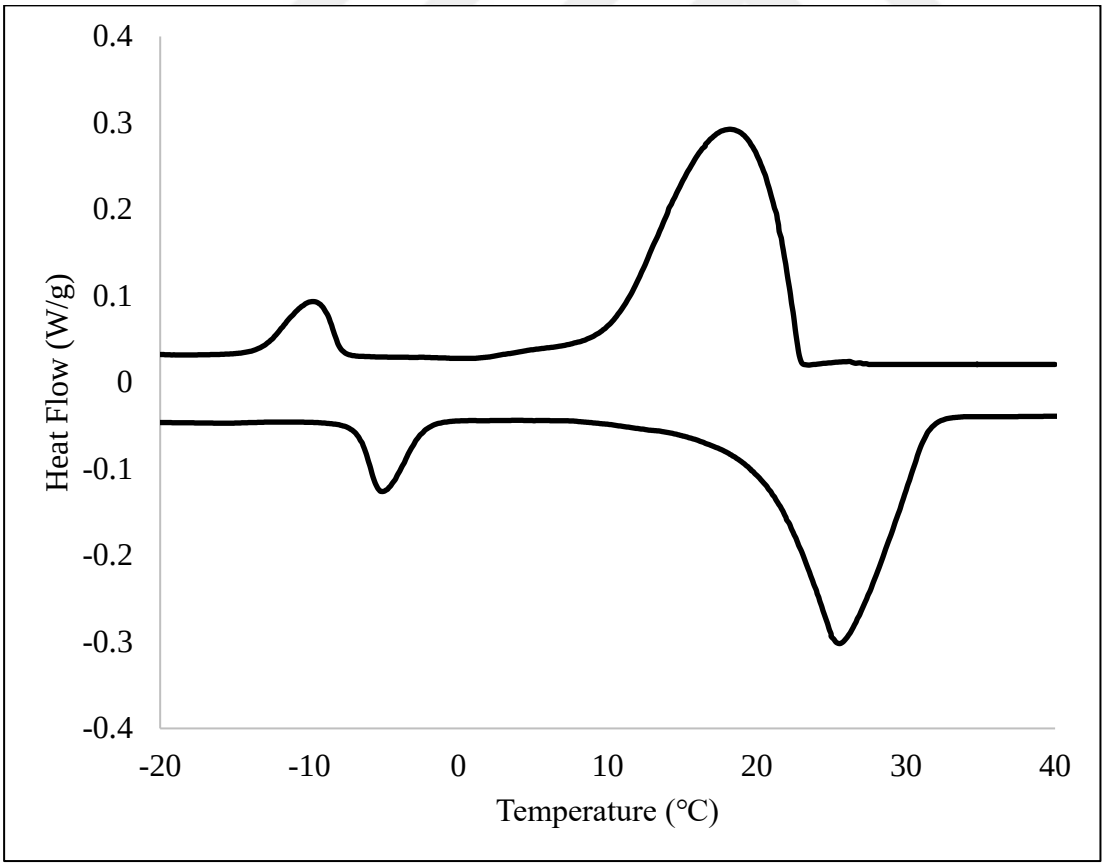


Figure 4.6 : Measured DSC curves of the PCM used in the study.

Table 4.5 : The results of the DSC analysis of PCM.

	Solidification process				Melting process			
	T_{onset} (°C)	T_{endset} (°C)	T_{peak} (°C)	ΔH_{exoth} (kJ/kg)	T_{onset} (°C)	T_{endset} (°C)	T_{peak} (°C)	ΔH_{endoth} (kJ/kg)
PCM	22.93	10.41	18.22	98.20	20.45	31.31	25.54	-90.79
Mix I (No PCM)	-	-	-	-	-	-	-	-
Mix II (4% PCM)	21.00	12.18	17.00	3.56	18.60	28.08	24.22	-3.45
Mix III (8% PCM)	25.46	13.00	18.12	6.85	18.92	30.89	25.12	-6.82

4.3.1.2 DSC analysis of the fiber reinforced PCM composites

Results of the DSC analysis of both PCM mortar samples containing and the plain ones are presented in Figure 4.7 as DSC thermographs. The diagram of the reference mixture (that contains no PCM) is a straight line because there is not any ingredient that has a serious phase change enthalpy. With the addition of the PCM, the phase change enthalpy of the mixtures was dependent upon the PCM amount used in composites. The details of the thermal parameters of the phase transition are presented in Table 4.5. Mix II (containing 4% PCM) completed its phase transition between 21 and 12.18°C with a peak temperature of 17°C during the solidification process. The enthalpy for this transition was calculated to be 3.56 kJ/kg. In the heating cycle, the phase transition started at 18.60°C and was completed at 28.08°C, with a temperature peak at 24.22°C. The absolute value of the enthalpy for this transition was calculated to be 3.45 kJ/kg, which is nearly equal to the value that was calculated for the solidification process. When the amount of PCM in the mixtures was increased from 4% to 8%, the phase transitions were observed at nearly the same temperature interval because the same PCM was used in all of the specimens. The phase transition enthalpy increased from 3.56 kJ/kg to 6.85 kJ/kg for solidification and from 3.45 kJ/kg to 6.82 kJ/kg for melting. Because the PCM is only material in the mixture that experiences phase transition in the studied range of temperatures and assuming that the enthalpy is linearly proportional to the proportion of the PCM amount in composites, the theoretical latent heat (ΔH_T) of the mixtures can be calculated as:

$$\Delta H_T = \Delta H_{PCM} \text{ x } W_{PCM}\,\% \tag{4.1}$$

where ΔH_T is theoretical latent heat, kJ/kg; ΔH_{PCM} is the measured latent heat of PCM, kJ/kg; W_{PCM} % is the weight percentage of PCM in the mixture [24], [148].

For Mix II, the theoretical latent heat values were calculated to be 3.93 kJ/kg and 3.63 kJ/kg for solidification and melting, respectively. The measured enthalpy values for Mix II were 3.56 kJ/kg and 3.45 kJ/kg for solidification and melting, respectively

(Table 4.5). The ratios of theoretical and measured values were calculated as 91% and 95% for solidification and melting processes, respectively. This finding indicates that the PCM in the GRCs performed as expected to enhance the thermal characteristics of the mixture.

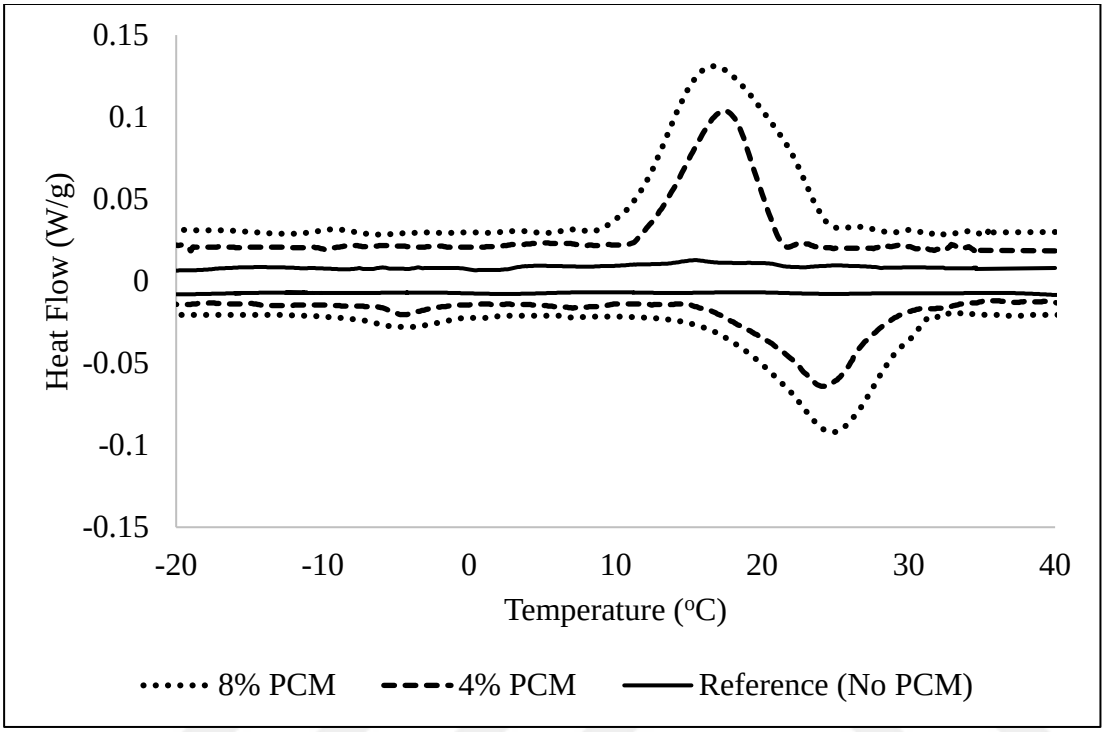


Figure 4.7 : DSC curves of the fiber reinforced PCM composites.

4.3.1.3 Thermal conductivity of the composites

Results of the conductivity measurements are summarized in Table 4.6. Although the change is not very drastic, the PCM addition caused a reduction of the thermal conductivity of the samples. The reduction is approximately 15% with respect to reference sample (Mix I), in the case of 4% PCM, and 24% for the samples that included 8% PCM. This reduction is attributed to the lower thermal conductivity of the paraffin-based microencapsulated PCM. When the results were analyzed (Figure 4.8), a strong relationship between thermal conductivity and PCM amount of composites was obtained and presented in (4.2):

$$\lambda = -0.0375\ W_{PCM} + 0.3583 \qquad (4.2)$$

where λ is thermal conductivity of fiber reinforced cementitious PCM composites, W/(m.K); W_{PCM} is the PCM percentage in the composite. The R^2 value is 0.9643.

This resulting empirical formula can be used to estimate the thermal conductivity values of GRCs enhanced with microencapsulated paraffin PCMs. Reducing the

thermal conductivity of the composites will increase the insulation of the buildings but it should be noted that, if the panels are intended to be used only for the energy storage, the lower thermal conductivity values may be a problem in terms of working efficiency [65].

Table 4.6 : Measured thermal insulation parameters of composites.

Mixture #	Thickness, h (mm)	Thermal transmittance, U [W/(m^2.K)]	Thermal resistance, R [(m^2.K)/W]	Thermal conductivity, λ [W/(m.K)]
Mix I (No PCM)	25	13	0.0769	0.33
Mix II (4% PCM)	25	11	0.0909	0.28
Mix III (8% PCM)	25	10	0.1000	0.25

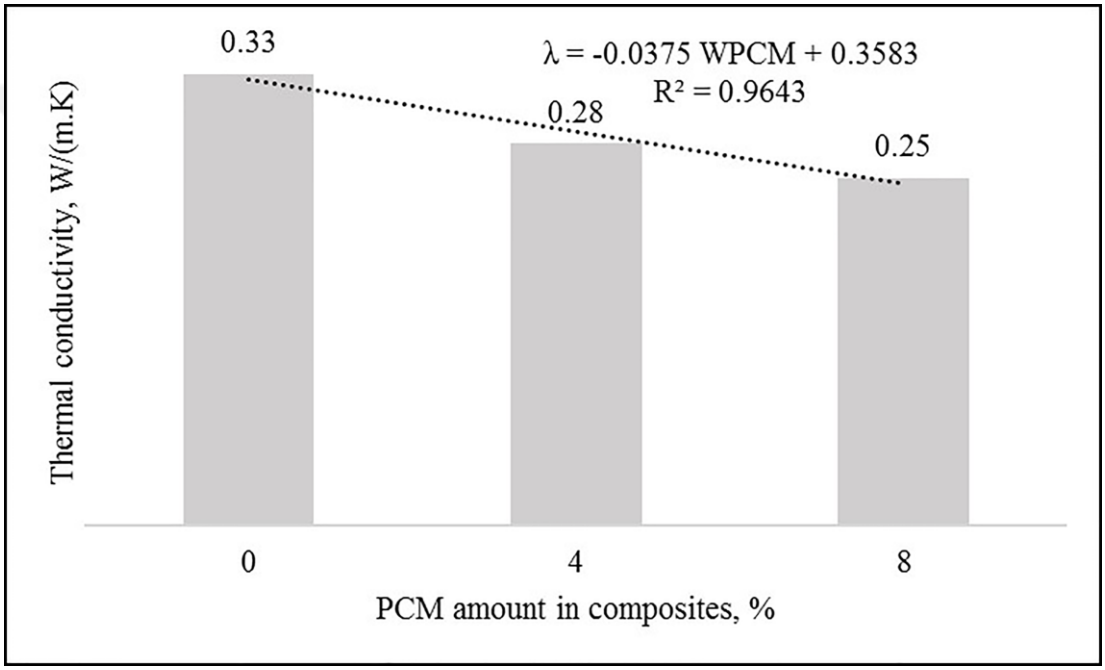
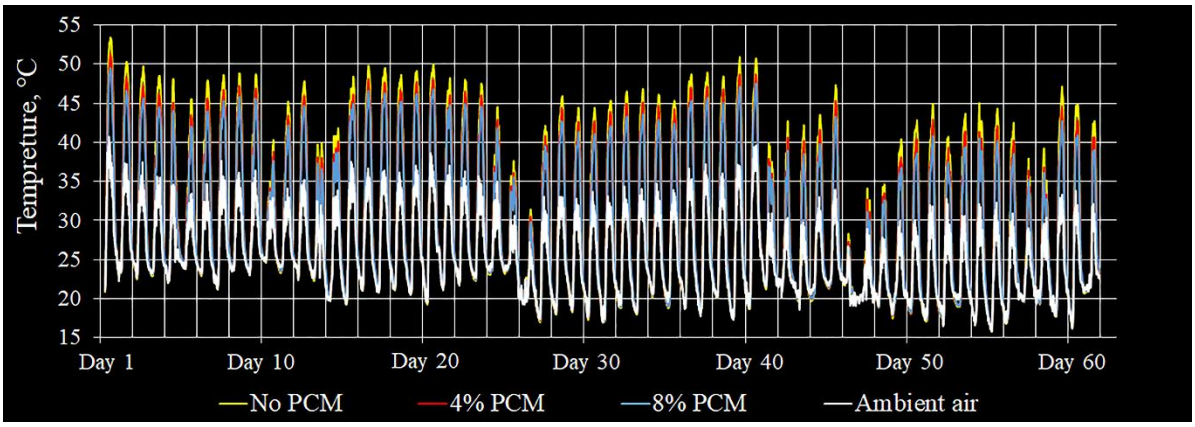


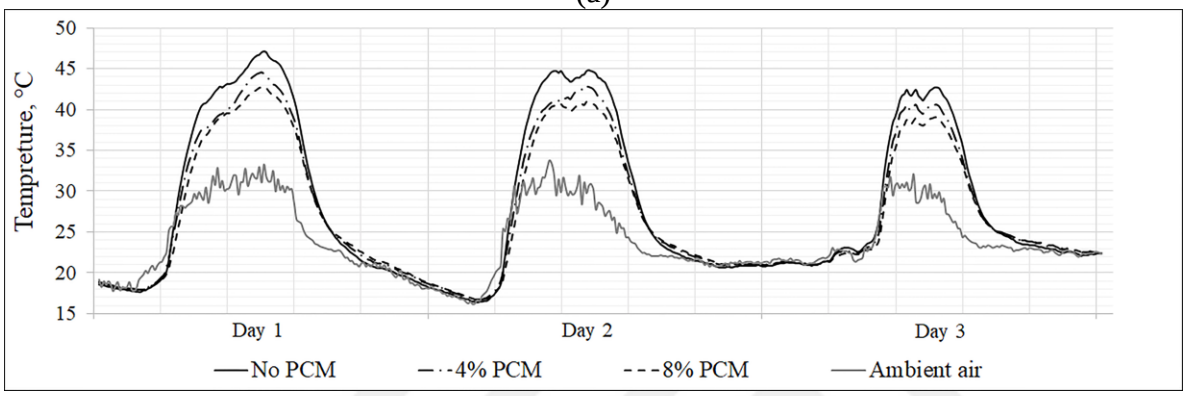
Figure 4.8 : Relation between thermal conductivity and PCM amount used in composites.

4.3.1.4 Temperature monitoring of the cubicles

The results of the temperature monitoring of the cubicles reveal the enhanced performance of the PCM composites and support the thermal analysis discussed above. In Figure 4.9 (a), the temperatures of the free-floating air inside the cubicles, as well as the air temperature are presented. The phase transition loop of the PCM was successfully repeated during the monitoring. The temperature variations among the cubicles can be seen in detail in Figure 4.9 (b), in which only three daily cycles are presented. The temperatures measured in cubicles during the day were higher than the air temperature as a consequence of the fact that the cubicles were built in a relatively small scale with no ventilation system.



(a)



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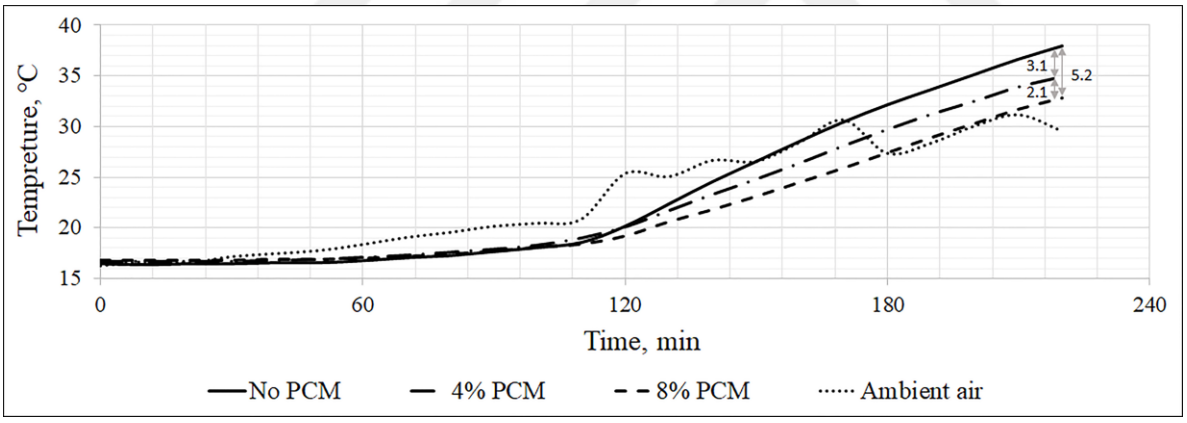

(c)

Figure 4.9 : (a) An overview of the temperature development under varying environmental conditions in summer days (b) Detailed view of temperature variations (c) Difference of temperature development in the cubicles following the phase transition of PCM within the composites.

The PCM in the cementitious composite affected the temperature development of the cubicle. The temperature peaks measured in the cubicle during the day decreased in comparison with the reference cubicle that contained no PCM. This behavior can be explained by the fact that the heat capacities of PCM composites are higher than those of plain composites. Another reason for this performance change is the relatively lower thermal conductivity of the PCM composites compared with the plain ones. These

features improve the insulation properties of the fiber reinforced composite. The temperature differences among the three cubicles were as high as 5.2°C in the case of 8% PCM compared with the plain one [Figure 4.9(c)]. The maximum temperature difference between the cubicle that contains 4% PCM and the one without PCM was 3.7°C. When Figure 4.9(c) is examined in detail, these differences can be attributed to the melting of the PCM inside the composite. The temperatures inside the cubicles started to vary from each other after the inside temperature reached approximately 18.5°C, which is the onset temperature of melting (Table 4.5). The reverse of this behavior also can be observed in Figure 4.9(b) as evening approached, when the temperature of cubicles begins to drop below 26°C which is the onset temperature of solidification (Table 4.5).

To evaluate the daily performance of the cubicles, the maximum temperature differences between the PCM cubicles and the plain cubicle were plotted (Figure 4.10) with reference to daily maximum air temperature. As observed from this figure, the thermal performance of the PCM-integrated fiber reinforced panels increased with increases of the difference between the inner and environmental temperatures. Figure 4.10 also shows that the PCM composites maintained their performance even at temperatures above their transition zone temperature. The relatively lower thermal conductivity of the composites can be shown to be the reason for this phenomenon.

As well as the promising contribution of the reduced peak temperatures during the day to the cooling and heating expenditures, another important issue is the time-shifting feature. This PCM feature would be more valuable in the countries where time-of-use (TOU) pricing is used. This would be especially valuable with the current policy of those countries that encourages the large-scale use of low-carbon heating systems that are required to be connected to the electrical distribution network by shifting the cooling load to a daily period with a lower electricity tax and thereby providing a great benefit to the economy [149], [150]. By changing the temperature acceleration [Figure 4.9(c)], fiber reinforced PCM composites will increase the thermal efficiency of a building and reduce the cooling expenses by removing the electrical loads of the cooling systems especially during the summer. Although no remarkable shift between the times that the cubicles reached their peak temperatures was encountered when the monitored data were analyzed, it is very clear that the PCM delayed the temperature increase inside the cubicle all of the time after the onset temperature of melting was

reached. The latent heat capacity of the PCM composites retarded the temperature increase inside the cubicle throughout the day. The cubicle containing 8% PCM reached its peak temperature 3 hours later than the plain cubicle.

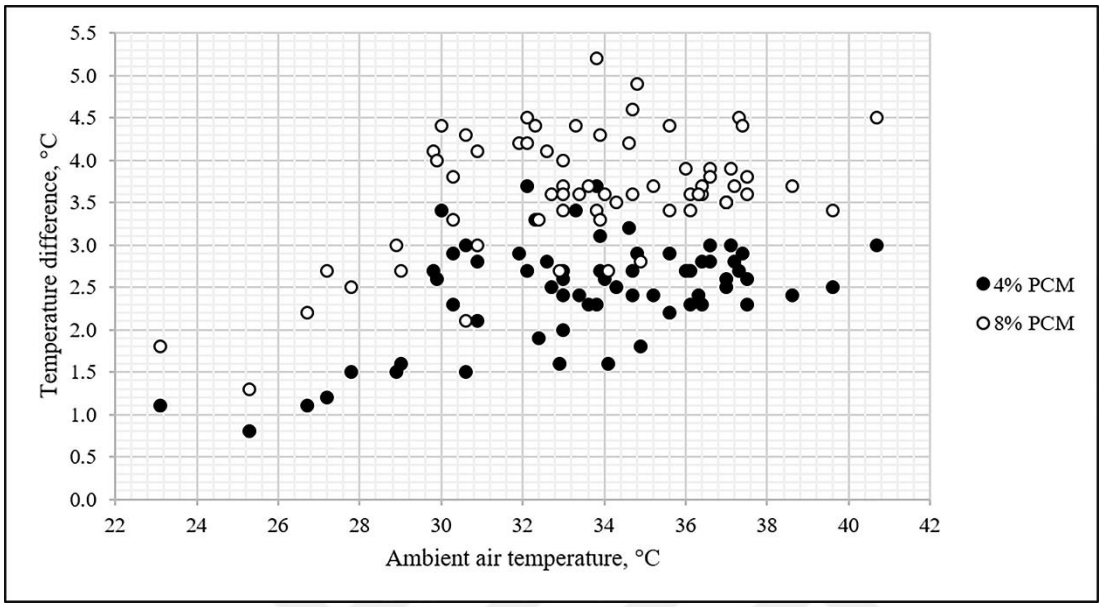

Figure 4.10 : Performance of PCM composites depending on the daily temperature fluctuations.

4.3.2 Mechanical tests

4.3.2.1 Compressive strength of the composites

The mechanical tests were conducted on two different sample sets conditioned at two different temperatures as explained above. The results of the uniaxial compressive tests are presented in Figure 4.11. In this figure, $\sigma_{10°C}$ and $\sigma_{50°C}$ are the compressive strength of FRC in MPa at 10°C and 50°C, respectively, and W_{PCM} is the PCM weight percentage of the composite. For both cases, the PCM negatively affected the compressive strength. The strength of the composite decreased with increasing PCM amount. While the average compressive strength of the mixtures without PCM was approximately 40 MPa, this value decreased to 21 MPa at 8% PCM. For the tests conducted at 10°C, the decrements of the compressive strengths were 5% and 30% for the 4% and 8% PCM composites, respectively. The decrements were 22.5% and 47.5% for the tests performed at 50°C. The reason for this decrease is attributed to the porous structure of the PCM composites in comparison with the plain cementitious matrix. The different physical state of PCM at the selected test temperatures also plays role in this drastic strength change. When the temperature of the specimens was raised above

the melting of PCM, the paraffin in the microcapsules transformed into a liquid from a solid. The encapsulated PCM particles in matrix may behave as voids at high temperatures. Mixtures lost 17.5% of their compressive strength independently of the PCM usage ratio when PCM transformed from a solid to a liquid. This phenomenon supports the idea that the physical condition of the PCM must be recognized and is crucial to the compressive strength of the material. However, even the lowest strength value obtained in this research can be considered to be satisfactory for the practical use of these composites in site applications.

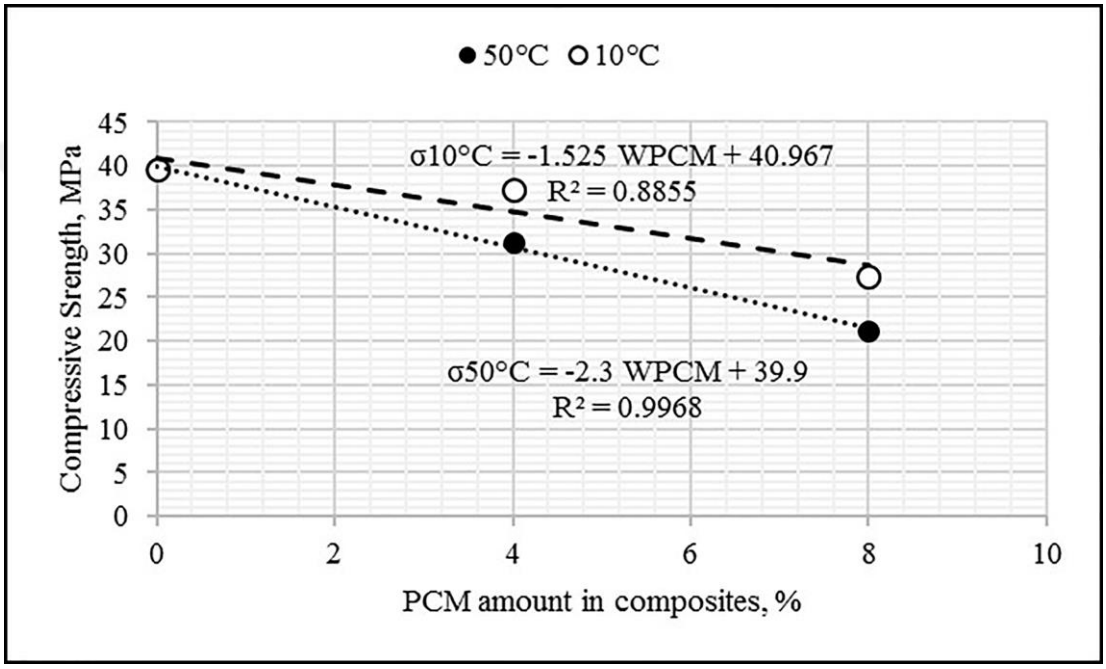

Figure 4.11 : Variation of the compressive strength by PCM amount and test temperature.

4.3.2.2 Flexural strength of the composites

Because all of the composites within this research have fiber reinforcement, the specimens have ductile behaviour under flexural loading. The typical behaviour of the specimen groups under the flexural loads can be observed in Figure 4.12. PCM incorporation enhanced the ductility of the composites for all of the PCM proportions used in this study. As seen in Figure 4.12, the flexural tests were also conducted at two different temperatures. The results of the flexural tests are presented in Figure 4.13. The flexural strength of the composites was negatively affected by PCM incorporation. The maximum decrement was 1 MPa. The maximum standard deviation between the individual results was 0.87. The PCM addition also affected the strain capacity of the mixtures (defined as the deflection at maximum load). In the tests conducted at 50°C,

the average strain capacity increased from 0.56 mm to 0.77 mm and 0.99 mm with the addition of 4% and 8% PCM, respectively. The strain capacity increased from 0.47 mm to 0.97 mm and 1.06 mm with addition of 4% and 8% PCM at 10°C, respectively.

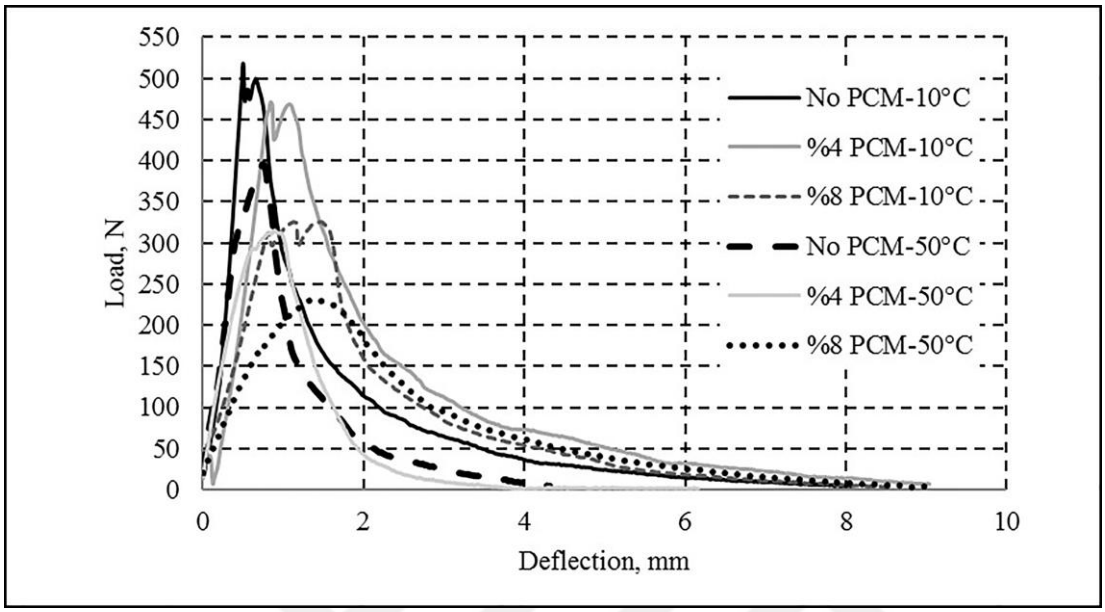

Figure 4.12 : Typical behaviour of mixtures under flexural loading.

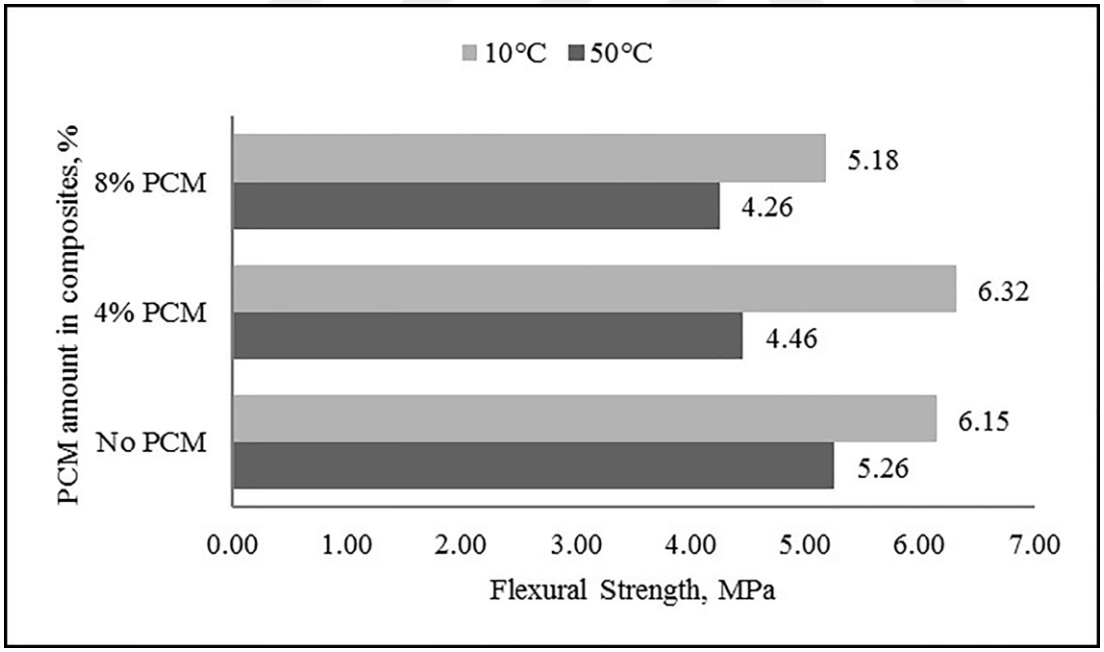

Figure 4.13 : Variation of flexural strength by PCM amount and temperature.

The test temperature affected the flexural strength of the specimens, even in the case of no PCM employment. At 50°C, decrements up to 29% were obtained compared with test results at 10°C (Figure 4.13). Because the flexural performance of fiber-reinforced boards is very important for a sustainable application, the designs for the

practical use of PCM composites, must take into consideration that at temperatures higher than the melting temperature of the PCM utilized in composites, a reduction of this performance criteria will be observed.

4.3.2.3 Fracture energy analysis of the composites

Fracture energy is the energy that is absorbed by the sample during its loading. The order of magnitude of this energy indicates the ductility of the material. The specific fracture energies (W_f) that were calculated based on the area under the specimen load-deflection curve are presented in Figure 4.14. Based on this figure, it can be concluded that PCM incorporation improved the energy absorption capacity of the fiber reinforced composites. A 4% PCM addition enhanced the specific fracture energy by 20%, while the improvement was 26% for 8% PCM addition for the tests conducted at 10°C. No systematic trends can be observed for the specific fracture energy values in the results of the tests conducted at 50°C. However, the energy absorption capacity was enhanced 51% by 8% PCM addition at 50°C. Although the fiber amount of the composites was the same for all of the mixtures in this study, the difference between the specific fracture energy values of the composites can be explained by the matrix properties. At the higher PCM proportion, the matrix strength was lower, allowing the fibers to be pulled out from the matrix resulting in a higher energy absorption value. On the other hand, in higher strength matrixes, fiber ruptures appear as a result of the complex interfacial characteristics between the matrix and the fibers, limiting the energy absorption capacity of the composite. This strong interfacial pattern also explains the higher flexural strength of plain mixtures compared with those incorporating PCM (Figure 4.13). It can be concluded that an optimization between the matrix strength and fiber amount is essential to reach the desired performance.

4.3.3 Discussion

PCM use in building envelopes brings several advantages both for residents and country economy. When considered more broadly, PCM has become one of the key terms of environmental progress with its contribution to sustainability. The main idea here is to be able to respond to a part of the building's energy needs by providing the energy storage feature to the existing elements of the building in addition to the building's own main functions and in cases where this is not possible, to be able to contribute to the insulation. There are many cases to be aware of when using PCM in

building elements. Although it is not possible to address all of these in a single study, it is worth pointing out the main points. When we consider human efforts to keep the interior temperature within a certain range in order to live a comfortable life, it is essential to select the melting / freezing temperature of the PCM, which is planned to be used in the construction, within the target comfort temperature range. The more the PCM containing materials of the building contribute to keeping the indoor temperature within this range, the more the load of the active air conditioning systems like air conditioners used at the buildings will decrease. Due to the different climates of different countries, effective PCM transition temperatures, which will maximize system efficiency, will also be different. Solgi et al. [96] investigates the financial viability of PCMs for different climates in Iran and summarized the change of the optimum PCM peak melting temperature for total annual energy consumption with different climate zones (Table 4.7) [96], [151], [152], [153]. The melting temperature of the microencapsulated PCM used in this study was also measured as 25.5°C in accordance with the values given in Table 4.7. Another study [154] offers to use PCM technology in the climates where the indoor temperature is mainly in the range of PCM transition temperature. For the climates where the indoor temperature mainly goes beyond the PCM functional temperature, the application of supplementary energy efficient techniques such as night ventilation were suggested [155], [156]. Factors such as gas tariffs, electricity tariffs, capital cost of PCMs, and bank interest also has a serious role on the economic assessment of the PCM utilization. Although the resultant payback periods change according to these factors, payback periods reported in literature are generally shorter than buildings average life spans [96], [97], [98]. When the results of the thermal experiments conducted in this study evaluated together, it is seen that the climate conditions of where PCM panels will be used is very important to obtain the best results and therefore the geographical location of the service area should be well analyzed before the design stage of the panels. Factors such as sunlight intensity, shadows that the structure exposed, daily temperature differences that the region experiences during the year can be shown as a few of the factors to consider. On a project basis, this technology can be used successfully after features such as PCM properties, ratio, panel thickness to be used are experimentally tested.

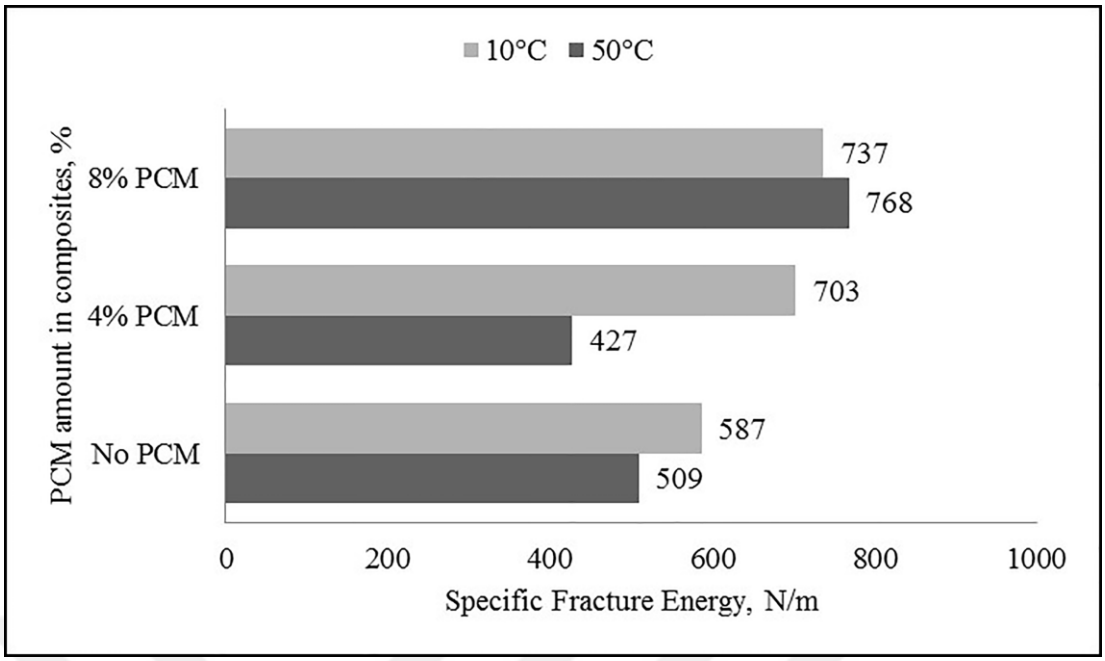

Figure 4.14 : Specific fracture energies of mixtures.

Another point that needs to be emphasized is the determination of the optimum PCM ratio to be used in the construction. In this study, it was observed that as the amount of PCM used increases, the thermal performance of the material increases in accordance with the studies in the literature [18], [24], [29], [46], [65], [66], [88], [92], [107], [135], [137], [138], [139], [140], [141], [142], [143], [144], [149], [150]. It can easily be said that when the amount of PCM is increased, the latent heat capacity of the mixtures increases and thermal conductivity decreases. On the other hand, when the PCM amount increases, the strength values of the building materials decrease considerably [66], [88], [143]. This phenomenon has limited the PCM use in the building materials such as mortar and concrete which contains hydraulic binders [150]. In our work, glass fiber was used for the preservation of mechanical properties and material ductility in cementitious PCM composites produced. With use of fibers, the decrease in the flexural performance was avoided to some extent comparing to the compressive performance. One of the important results related to the use of PCM in cementitious composites obtained in the study is the severe decrease in mechanical performance with the transition of solid phase of PCM used in the material to the liquid phase. In the literature, the results supporting this situation have been reported [150]. The fact that the PCM in the matrix behaves as a void with this transition and the material structure which now becomes more porous and permeable, can be shown as the reason for this situation. For a safety-wise solution, the service temperature range

of composites to be produced using PCM should be determined carefully and realistically, and the performance changes in composites should be fully revealed by carrying out experimental studies considering the most adverse conditions before use.

4.4 Conclusions

In this study, the use of microencapsulated PCMs in fiber reinforced cementitious composites was investigated, and the thermal and mechanical performances of the fiber reinforced PCM composites were studied. Composites containing as much as 8% microencapsulated PCM were produced and characterized. The conclusions obtained from this experimental work can be drawn as following:

PCM incorporation increased the thermal storage property and thermal mass to the composite material stepwise with the PCM proportion. The enthalpy of the mixtures increased gradually with the PCM proportion and the enthalpy values obtained by the tests closely matched the theoretical latent heat values of the mixtures. On the other hand, the thermal conductivity of the composites decreased as the PCM amount in the mixture increased.

The temperature differences between the plain cubicle and the PCM cubicles were as high as 5.2°C during the day. The PCM composites exhibited a successful thermal performance limiting the excessive heating of the cubicles. When the energy storage property of PCMs is taken into consideration, this accumulated heat can be employed at night when the temperature falls under the PCM solidification temperature in the regions with compatible climate, leading to the efficient use of energy.

The latent heat capacity of the PCM composites delayed the temperature increase inside the cubicle throughout the day. Shifting the time of the peak temperature to another time zone with a lower tariff would contribute to energy costs savings.

In the second part of this research, mechanical tests of the composites were conducted to evaluate the structural performance of the mixtures. Because the main objective of this study was to evaluate the mechanical performance characteristics of the PCM composites, tests were performed on samples at different temperatures. Within the limits of the work conducted in this investigation, it was observed that PCM addition decreased the uniaxial compressive strength by as much as 47.5% when the temperature of the composite exceeded the melting temperature of the PCM. Mixtures

lost 17.5% of their compressive strength independently of the PCM usage ratio when PCM transformed from a solid to a liquid. The flexural strength of the composites was also negatively affected by PCM incorporation by up to 29%. On the other hand, the PCM enhanced the ductility of the composites by changing the interfacial characteristics between the matrix and fibers. Both the energy absorption and the strain capacity of the composites were increased by PCM addition. In conclusion, microencapsulated PCM glass fiber reinforced concrete is a promising material for sustainable buildings. These composites can reduce building energy consumption and increase thermal comfort. But for a safe use, considering the fluctuations at different service temperatures, the performance should be adjusted in such a way that it does not fall below the design strength value.

5. CONCLUSIONS AND RECOMMENDATIONS

Alternative methods of utilizing PCMs in building materials were investigated within this dissertation. In the first two articles (Chapter 2 and Chapter 3), a sustainable cold bonded lightweight PCM aggregate production and its effects on concrete properties were discussed while mainly concentrating on the design, production and performance of the pelletized aggregates. In the third article, which was explained in Chapter 4, main subject is the thermal and mechanical performance of GRCs containing microencapsulated PCM. This chapter specifies the main contributions, some of the blank and blind spots of the conducted research and delineates perspective on future directions.

The study that focuses to the design, production and performance of artificial aggregates showed that each material included in the mixture affects the water required for pellet formation. This phenomenon reflected on the general results of the study directly, affecting the pelletization process and the engineering properties of the product. Modifications in the pore structure of the produced aggregates arising from different water contents of the mixtures, directly affected both the physical properties of the aggregates such as density and permeability and strength properties such as crushing strength. Consequently, in future works, the aggregate designs should be shaped with trial productions according to the ingredients. The effect of sand usage on both the pelletization process and aggregate properties was another subject investigated in this part of the research. It was found out that sand facilitates pellet formation and accelerates the process. On the other hand, when the amount of sand in the mixture passes a certain threshold, the amount of binder in the mixture becomes insufficient to bind the whole content and this leads to the accumulation of unbound material in the base of the pelletization disk. Another finding related to the sand is that the sand causes some increase in the density of the aggregates produced. Besides, it does not generate a significant increase in the strength of the aggregates. After all, it can be concluded that keeping sand usage to a minimum level to facilitate pelleting will be the optimum solution. Based on the Taguchi and variance analyses, binder type

was determined as the most effective parameter on the physical properties such as pellet density, water absorption and permeable porosity. In the case of strength properties, curing duration was found as the most effective factor. It was also obtained from these analyses that the interaction of the binder type and the curing duration is also highly effective on the strength. The study was also enabled the comparison of aggregates produced using different binder types such as cement, FA and GGBS, and it showed that the aggregates produced using GGBS are lighter in proportion to the aggregates produced using FA. When the strength properties of the aggregates are considered, it can be concluded that although the early strength of GGBS aggregates is higher than FA aggregates, with the prolongation of curing duration, FA aggregates show a better performance in terms of final strength.

In the next phase of the study, aggregate designs were developed by adding microencapsulated PCM to the mixture, and it was investigated whether artificial aggregates containing PCM could be produced by using pelletization method. This part of the study exposed the effects of PCM both on the pelletization process and properties of aggregate products. Although PCM negatively affected the pelletization process, it provided an increase in the thermal performance of the aggregates. In addition, by means of PCM, lighter aggregates were obtained, which supported the lightweight feature, one of the most important artificial aggregate production objectives. It was seen that the PCM incorporation decreased the particle density gradually down to 0.99 g/cm^3. The study also introduced the micro structural differences arising from the differences in the water amount of pellets, the effects of binder composition and PCM use. Mechanical properties of aggregates were degraded regardless from binder type in case of PCM addition. It was found out that the effect of PCM ratio on crushing strength of pellets is more significant than effect of binder type. A high correlation between the crushing strength and aggregate density was also detected. Based on the obtained results, it can be concluded that, in general, as the particle density of the aggregates decreases based upon the increase of PCM ratio of the mixtures, the crushing strength of the aggregates also decreases. When the thermal performance of the produced PCM aggregates was evaluated, it was seen that the different concerns such as strength, producibility and cost efficiency limit the PCM ratios of the aggregates, hence the TES performance of the material. Another point to be emphasized here is the thermal testing process of the materials. Along with the

reasons rising from the material structure, inconveniences encountered in production and test sample preparation stages also affect the test results, masking the real performance. On the other hand, micro-scaled samples weighed in mg level, decrease the chance of making a homogenous sampling that represents overall aggregate, especially in relatively low amounts of PCM containing mixtures. It is therefore difficult to take consistent results via DSC tests that reflect the macro properties of composite aggregate. As future work, LWAs with high porosity may be produced by pelletization method and after a desired strength development is achieved, a better thermal performance can be obtained by PCM impregnation method. In this way, sintering or hydrothermal treatment can be applied to aggregates to provide them extra strength prior to the PCM impregnation process. Aggregate surfaces may be coated with epoxy or modified cement paste to prevent PCM leakage during phase change process. Another useful research topic may be the thermal characterization of real scale building materials as the next strand of the research. Considering that the generated aggregates are not intended to be used for direct energy storage, it can be concluded that these LWTAs will contribute to thermal comfort by blocking excess solar energy from outside the building, within the concept of passive construction. Concretes including produced LWTAs were also produced and their engineering properties were examined. Experimental results showed that lightweight concretes can be produced by using LWTAs produced within the study. Based on the experimental results, it was seen that although the PCM causes a decrease in compressive strength of the concrete, it is still possible to produce a structural concrete with a sufficient strength for many structural applications.

In the last part of the research, GRC panels containing microencapsulated PCM were studied. By the direct incorporation of the microencapsulated PCM into the GRCs, a remarkable improvement was achieved in the thermal performance of composites. The study showed that the PCM in the cementitious composite affects the indoor temperature development successfully by blocking the excessive heat coming from the sun. Advanced heat capacity of PCM composite together with its relatively lower thermal conductivity improve the insulation properties of the material. It also changes the temperature acceleration and enables shifting the time of the peak temperature to another time zone which offers a lower tariff. By this feature, the initial investment in PCM-containing building materials will easily pay for itself in the oncoming years and

these materials will lead the building owner to profit. In addition, the PCM composites maintain their performance even at temperatures above their phase transition temperature zone. In conclusion, fiber reinforced PCM composites will increase the thermal efficiency of a building and reduce the cooling expenses by removing the electrical loads of the cooling systems especially during the summer. In addition to the improvements in thermal performance of the material, the study focused on the extent of the mechanical performance loss and questioned the change in mechanical performance after the phase transformation of the PCM within the GRC. It was obtained that the decrease in uniaxial compressive strength of composites is higher than the decrease in flexural strength.

In conclusion, cementitious PCM composites is a promising material for sustainable buildings. These composites can reduce building energy consumption and increase thermal comfort. However, for a safe use, the performance should be adjusted in such a way that it does not fall below the design strength value, considering the fluctuations at different service temperatures. Future research can be directed towards investigating the economic feasibility of the cementitious composites including PCM, which is out of the scope of this dissertation. Experimental works including real-scale tests can also lead to a better understanding of the issue.

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