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PREPARATION OF SILICA NANOCAPSULES CONTAINING PHASE CHANGE MATERIAL

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
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THE DEGREE OF MASTER OF SCIENCE
IN
CHEMICAL ENGINEERING

BY

ABOUBAKER-SADIK EGUEH IYEH

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PREPARATION OF SILICA NANOCAPSULES CONTAINING PHASE CHANGE MATERIAL

By Aboubaker-sadik egueh IYEH

August 2023

We certify that we have read this thesis and that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science

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ABSTRACT

PREPARATION OF SILICA NANOCAPSULES CONTAINING PHASE CHANGE MATERIAL

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Silica nanoparticles (SNPs) can be produced in small diameters with good reproducibility, stability, and biocompatibility for carrying phase changing materials (PCM) to develop a new formulation for heating and cooling applications. In this work, a capsule-like silica structure was obtained to carry nonadecane as a PCM and characterized by dynamic light scattering (DLS), zeta potentials, scanning electron microscopy (SEM), Fourier-transform Infrared (FTIR) and differential scanning calorimetry (DSC). Classically, SNPs are synthesized by Stöber method as empty formulations. To achieve a capsule-like structure with small sizes, we used a modified technique including an oil phase containing dissolved PCM addition to the water phase at lower temperatures. This method ensures a slower and controlled reaction which allows smaller particle sizes. SNPs were synthesized in two types as nanodecane loaded and empty as a control. After the synthesis, characterization steps were performed. First of all, particle size was checked by DLS and found as 50 nm. Zeta potentials were also measured as -30 mV'dir. Which suggests the SNP formulation is stable in water. SEM images showed that particles are synthesized successfully with a spherical shape. Also, SEM images confirm the measured nanoparticle size by DLS. FTIR showed that SNPs were synthesized with the characteristic peaks at 1100, 964 and 796 cm -1. According to DSC results, nonadecane was loaded into SNPs confirmed by the peaks around 25-40 o C while the empty formulation showing no peaks. These results show that nanodecane loaded SNP is a good candidate for PCM applications such as building heating and cooling or heating pads.

2023, 58 pages

Keywords: Phase changing materials (PCM), Silica nanoparticles (SNP)

ÖZET

FAZ DEĞİŞTİREN MADDE İÇEREN SİLİKA NANOKAPSÜLLERİN HAZIRLANMASI

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Silika nanopartiküller (SNPs), ısıtma ve soğutma uygulamalarında kullanılabilecek bir formülasyon elde etmek üzere faz değiştiren madde (PCM) taşımak için küçük partikül büyüklüklerinde, tekrarlanabilir, stabil ve biyouyumlu olarak üretilebilirler. Bu çalışmada, kapsül benzeri silika yapısı, bir PCM olan nonadekanı taşımak üzere elde edilmiş ve dinamik ışık saçılımı (DLS), zeta potansiyeli, saçılımlı electron mikroskopisi (SEM), Fourier- dönüşümlü kızılötesi (FTIR) ve diferansiyel taramalı kalorimetri ile karakterize edilmiştir. Klaik olarak, SNPler Stöber yöntemiyle içlerine madde yüklenmeden sentezlenirler. Küçük boyutlu kapsül yapısı elde edebilmek için görece düşük sıcaklıklarda çalışılan, içinde PCM çözündürülmüş yağ fazının su fazına eklenmesini içeren modifiye bir Teknik kullanılmıştır. Bu metot, daha yavaş ve kontrollü bir reaksiyon sağladığından, daha küçük partikül büyüklükleri elde edilir. SNPler boş ve dolu olmak üzere iki formülasyon şeklinde üretildi, boş formülasyon kontrol olarak kullanıldı. Sentez sonrası, karakterizasyon çalışmaları yapıldı. Öncelikle DLS ile partikül büyüklüğü 50 nm olarak ölçüldü. Zeta potansiyelleri -30 mV olarak ölçüldü ki bu da SNPlerin su içinde stabil olduğunun bir göstergesidir. SEM görüntüleri partiküllerin küresel olarak başarılı bir şekilde sentezlendiğini gösterdi. Ayrıca, SEM görüntüleri DLS ile elde edilen partikül büyüklüğü sonuçlarını da doğrulamıştır. FTIR, SNPlerin 1100, 964 ve 796 cm-1’deki karakteristik piklerini göstermiştir. DSC sonuçlarına göre boş nanopartiküller pik vermezken nonadekan yüklü SNPler 24-40oC arasında pik vererek nonadekanın SNPlere yüklendiği gösterilmiştir. Bu sonuçlara göre nonadekan yüklü

SNPler, binaların ısıtılıp soğutulması veya ısıtma pedleri gibi başka PCM uygulamaları için uygun bir aday olabilir.

2023, 58 sayfa

Anahtar Kelimeler: Faz değiştiren maddeler (FDM), silika nanopartiküller (SNP)

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Aboubaker-Sadik Egueh IYEH
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CONTENTS

LIST OF SYMBOLS

-	Minus
%	Percent
/	Division
+	Plus
<	Less than
>	Greater than
°C	Degrees Celsius
Cp	Specific heat capacity
FG	Gravitational force
Keff	Effective thermal conductivity
Knp	Nanoparticle thermal conductivity
Kp	Equilibrium constant
ΔH	Enthalpy
ΔT	Temperature differential

LIST OF ABBREVIATIONS

AFM	Atomic Force Microscopy
Al-CaO3	Alginate calcium carbonate
DLS	Dynamic Light Scattering
DSC	Differential Scanning Calorimetry
EDX	Elemental distribution analysis
ES	Energy storage
FTIR	Fourier Transform Infrared Spectroscopy
LHS	Latent heat storage
NH3	Ammonia
Nm	Nanometer
NPs	Nanoparticles
OS	Octylsilane
PCM	Phase Change Material
PEG	Polyethylene Glycol
pH	Potential of Hydrogen
PVA	Polymethyl alcohol
SDS	Sodium dehydrocysulfate
SEM	Scanning Electron Microscopy
SHS	Sensible heat storage
SiO2	Silicon Dioxide
Si-PDMS	Polydimethylsiloxane
Si-UN	Unmodified silicat
SLPs	Sentinel lymph nodes
SSS	Sodium silicat solution
TCHS	Thermal chemical heat storage
TEM	Transmission Electron Microscopy
TEOS	Tetraethyl Orthosilicate
TES	Thermal Energy Storage
TGA	Thermogravimetre analysis
TMSPM	3(methoxycul propyl methacrylate

TPU	Thermoplastic polyurethan
XPS	Xray photoelectron spectroscopy
XRD	X-Ray Diffraction

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1. INTRODUCTION

Energy has an impact on human civilization's forward movements (X. Yang *et al.* 2021). However, energy crises has become a serious global issue due to rising energy consumption (Poudyal *et al.* 2019). Consequently, increasing energy efficiency and creating alternative energy sources is currently a pressing concern (Akbari *et al.* 2001).

The term thermal energy storage (TES) refers to any system that makes use of the heating or cooling of a specific material to store thermal energy (Sarbu *et al.* 2018). This makes it possible to store energy for later use in power generation, heating, and cooling applications (Lipka *et al.* 2020). There are several types of thermal energy storage such as storage of sensible heat, whose role is to store energy by changing the temperature of the material without renewing the phase (Singh *et al.* 2022). Additionally, there is latent heat storage, which utilizes latent heat to store energy through phase modification (PCM) between the solid and liquid phase. (Alawadhi, 2008). Energy can also be stored by thermochemical means, which encourages reversible chemical processes (Prieto *et al.* 2016).Phase change material (PCM) storage and thermoacoustic storage are two alternative methods of storing thermal energy (Liu *et al.* 2020)**.** Thermal energy storage has a variety of uses, including in industrial operations, HVAC systems, and the control of a building's energy use (Arteconi *et al.* 2017). Other application areas include the integration of renewable energies, power generation and grid stability, and district heating and cooling (Nguyen *et al.* 2021).

Phase Change Materials, often known as PCMs, are substances that, when heated to a certain temperature, experience a change in phase, generally changing from a solid to a liquid or vice versa (Singh *et al.* 2018). When PCMs go through this phase change, they are able to either store or release a large amount of thermal energy in the form of latent hea**t** (Pasupathy *et al.* 2008).There are the different types of phase change materials the Paraffin wax, Salt hydrates, Organic PCM and Inorganic PCM (Dinker *et al.* 2017). One type of phase change material is called a phase change silicate, these silicates often go through a reversible phase transition, such as melting or solidification, and technique that involves significant heat absorption or output (Hammonds *et al.* 1996). Due to their ability to store energy, phase change materials are finding applications in areas such as building and construction, electronics cooling, solar energy systems and automotive and aerospace (J. Huang *et al.* 2021). Encapsulating phase change materials refers to the act of enclosing the PCM within a protective or containment structure in order to improve the material's stability as well as its thermal properties and overall performance (Shchukina *et al.* 2018). Encapsulation helps to prevent PCM leakage, enhance thermal conductivity, permit regulated release of the stored energy, and protect the PCM from external variables and Encapsulation also helps to improve thermal conductivity (Cheng *et al.* 2022). The different types of encapsulation are macro, micro and nanocapsulation each of which has specific methods for their synthesis, the methods can be physical or chemical (Suresh *et al.* 2022).

Loading silicate nanoparticles in FDM 3D printing largely serves to improve the produced components' mechanical qualities and thermal stability (Ahmed *et al.* 2020). Scanning and transmission electron microscopy (TEM and SEM) use electron beams transmitted through a sample to obtain detailed three-dimensional images of the sample's structure, topography, and composition (McCutcheon *et al.* 2015). Differential scanning calorimeter **(**DSC) is a thermal method that measures the temperature differential between a sample and a reference material in addition to the heat flux melted at the phase transition (Zamani-Babgohari *et al.* 2023). The desired outcomes aimed to be achieved in this thesis proposal, which focuses on the preparation of particles encapsulating phase-change materials (PCM) by using silicat nanoparticles (SNPs), can be summarized as follows:

- During the synthesis of SNPs, the encapsulation of PCM is intended within them.

- In the preparation of SNPs, enhancement of PCM loading capacity, and optimization of particle size, an experimental design methodology is aimed to be employed to efficiently determine the optimal formulation through a limited number of experiments.

1.1 Thermal Energy Storage (TES)

Most thermal energy is stored as changes in the temperature (sensible heat storage) or the phase (latent heat storage) of a medium (which can be a fluid, a solid, or a combination of the two), and TES is the term used to describe this process. (Hedengren *et al.* 2014). Thermal energy storage has recently garnered increased attention and is therefore one of the most essential energy technologies (Acar *et al.* 2014).

The energy storage method known as TES has been around for a long time since it employs one of the earliest kinds of energy storage. Its ease of use has made it a viable energy storage option. When compared to storage methods like battery storage, which rely on expensive and sometimes exotic materials, TES is a relatively cost-effective way of energy storage (Powell *et al.* 2016).

Integration of more advanced energie storage (ES) systems with other technologies can produce innovative solutions that are more practically applicable in the ES areas and It is possible to divide ES devices into many groups (Gallo *et al.* 2016). There are many different ways to store thermal energy, and each one is tailored to meet a different set of requirements (Soares *et al.* 2014).

The following are some of the approaches that are most frequently utilized. (Table 1.1)

Table 1.1 Classification of energie storage method (Krishan, 2019)

Energy storage method		
Mechanical	Chemical	Others
-Hydrostorage (pumped storage) -Compressed air storage -Flywheels	-Electromechanical batteries -Organic molecular storage	-Biological storage -Magnetique storage

The fact that the process may move from liquid to solid, from gas to liquid, and from gas to solid, there is really a conversion of energy that takes place throughout phase shifts, the numerous criteria can be utilized to classify solutions storage technologies (Díaz-González *et al.* 2012). (Figure 1.1) Depending on the material's condition, various energy storage options are categorized and The most common approach of heat storage is known as sensible heat storage (SHS), it's the simplest and most fundamental method of thermal storage currently available (Sarbu *et al.* 2019).

Sensible heat is the heat exchanged by a system that maintains its phase but alters the storage medium's temperature (P. K. S. Rathore *et al.* 2022). Liquid salt, paraffin wax, water and ice cubes are the most common substances used for latent heat storage (LHS), which is the transfer of heat produced by a phase change that happens within a limited temperature range in the relevant material (Elias *et al.* 2019).

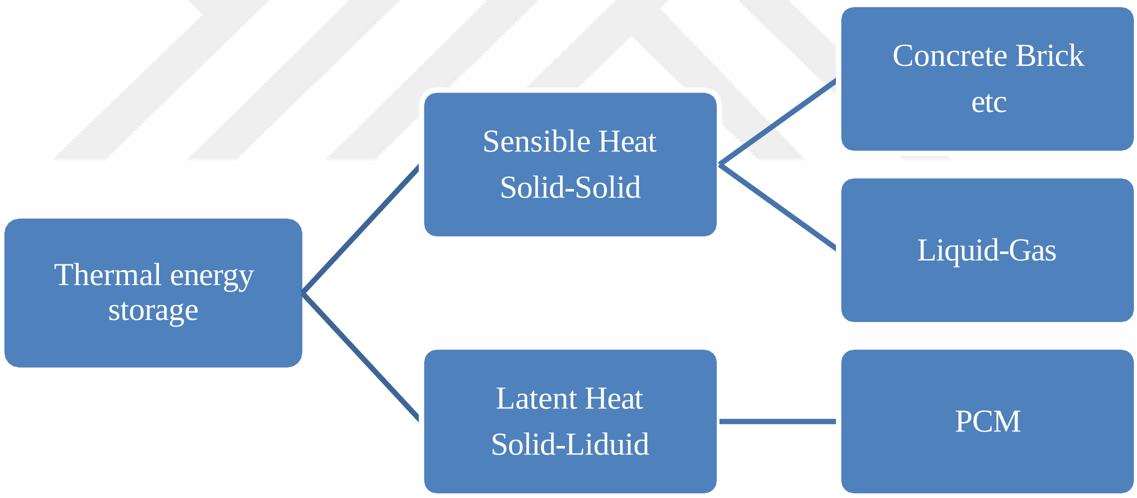


Figure 1.1 Type of thermal energy storage (Socaciu, 2012)

Analyzes the range of TES systems by contrasting their average energy densities to find out what features they share and what sets them apart. (Ibrahim *et al.* 2008). Furthermore, the scientific literature suggests that PCMs are capable of storing up to two to five times more heat than SHS materials(Shalaby *et al.* 2014). Many PCMs have a lower specific heat compared to water, which serves to reduce the discrepancy in energy density despite the fact that PCMs use the latent heat value however TCM's heat storage density can be anywhere from five to 10 times that of a SHS (Yinping *et al.* 1999). This difference is dependent on the kind of chemical reaction that occurs during the adsorption process, which can either be physical or chemical. The table 1.2 below shows the characteristics of SHS, LHS and TCHS in the various sectors.

Table 1.2 Comparing characteristics of different TES systems (Desai *et al.* 2021)

Thermal energy storage			
Characteristics	SHS	LHS	TCHS
Energy storage density	Small (0.18 GJ-m³)	Moderate (0.36 GJm")	High (1.8 GJm-3)
Heat storage value	$mCp\ \Delta T_{SHS}$	mHp	$F_f\ m\Delta H_{TCM}$
Heat storage dependency	Increase of temperature	Heat of fusion	Enthalpy of reaction
Temperature of storage	Final charging temperature	Low Final charging temperature	Ambient temperature
Duration of storage	Depending on the heat loss	Depending on the heat loss	Theoretically unlimited
Heat transport distance	Short	Short	Long
Maturity of technology	Commercialized	Pilot scale	Laboratory scale
System complexity	Low	Low	High
Heat loss during storage	High	High	Minor
Insulation requirement	Yes	Yes	No
Cost	Low	Moderate	High

Each thermal energy storage system has its own specific application, so it can play a crucial role in their development and advancement. Due to its simplicity and low cost, thermal energy storage (TES) is utilized in solar water heating systems, air heating systems, and kitchens. It can also be used in the refrigerator because it maintains a constant temperature on uncovered objects in the refrigerator. Other areas, such as PCM trompe wall, solar green house, and defense, can also come into action (Figure 1. 2).

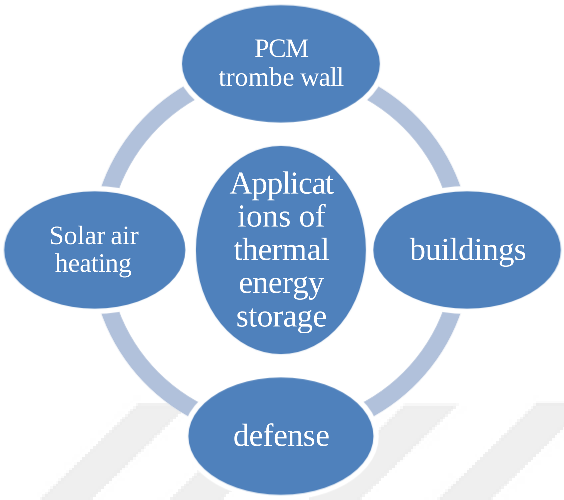


Figure 1.2 Classification based on application (Ali *et al.* 2020)

1.1.1 Phase change materials (PCM)

PCM has specific properties which are based on the storage of thermal energy, it allows to transform the storage per unit of volume greater compared to the energy of the material of conventional construction when the ambient temperature begins to increase (Paneri *et al.* 2019). There are many uses for phase change materials (PCMs) due of their many benefits (Oró *et al.* 2012).İt has a high latent heat which gives it the ability to not only store but release a significant amount of energy during phase transition (Mishra *et al.* 2015). The absorption and release phenomena are as follows, PCM absorbs energy when it melts and releases energy when it solidifies, thanks to this phenomena it becomes very essential for thermal energy systems (Chriaa *et al.* 2020). The PCM plays very important role in temperature regulation because it can maintain a constant temperature throughout the transition (Mohammed *et al.* 2021).

This characteristic is specifically advantageous in applications where temperature control is difficult, such as building insulation, cooling of electronic devices (Guarino *et al.* 2017). İt also develops the thermal comfort in the building to maintain a very stable lower temperature (Evola *et al.* 2013). The result may be reduced electricity costs and less harm to the environment (Al-Yasiri *et al.* 2022).Phase-change materials are also used in the industrial and solar energy sectors (Table 1.3).

Table 1.3 Applications of phase change material (Krishna *et al.* 2017)

Application of phase change materials	
Electronic	Thermal management of electronic devices (cell phones and computers).
Food industrial	Energy savings in the drying process and temperature distribution of foodstuffs
Building	Good energy storage capacity for building materials and water heaters
Solar energy	Night-time use of geothermal heat pumps

PCMs may survive several phase changes without significantly degrading in performance, giving them a long cycle life (Reed *et al.* 2019). They maintain their integrity during repeated melting and solidification, making them suitable for long-term usage (Li *et al.* 2012). There are many different kinds of phase change materials (PCMs), and they all have their own special properties and uses. Some typical examples of phase transition materials are listed below (Table 1.4) (Dutkowski *et al.* 2020).

- Organic materials, which make up several chemical compounds such as paraffin, fatty acids and organic mixtures and other elements (Meng *et al.* 2022).
- Inorganic PCMs can be divided into two categories such as salt hydrates and metals, they have a greater thermal energy storage capacity (approximately twice that of organic SL-PCMs) (Ghasemi *et al.* 2022).

- For eutectic phase change materials, both organic and inorganic compounds are required (Capric-laurie, Lauric palmitic, Myristic-stearic), as well as inorganic and inorganic compounds (LiOH-KOH, MgKCl2-KCl, BACl2-KCl, NaCl) (Sun *et al.* 2023).

Table 1.4 Classification of PCM (Rathore *et al.* 2019)

Phase change materials		
Organic	İnorganic	Eutectic
- Fatty acids - Paraffin - Ester - Alcohols	- Salts - Salt hydrate - Metal	- İnorganic-inorganic - Organic-organic - Organic-inorganic

1.2 Encapsulation

By definition, encapsulation is a technique in which one or more mixtures of materials are trapped inside other materials, and the coated materials are often liquids but can also be solids or gases (Risch, 1995)**.** It is also a method which aims to avoid or prevent direct contact between the PCM substance and the external environment in order not only to reduce the risk of leakage but also to increase the lifetime by reducing the problem associated with separation (Javed *et al.* 2021). Improving the efficiency of this method (encapsulation) involves increasing the surface area available for heat transmission initially, then using nano-additives which in turn increase conductivity, In other wise, these two factors have a considerable influence on the encapsulation phenomenon (Han *et al.* 2022). There are essentially several encapsulation methods, such as physical, chemical and physico-chemical, each of which has its own principles (Table 1.5).

Table 1.5 Encapsulation methods (Podara *et al.* 2021)

Encapsulation methods		
Physical	Chemicals	Physical-chemicals
- Spray dyring - Spray cooling - Fluidized bed - Coating	- Emulsion and Mini emulsion polymerization - İnterfacial polymerization - condensation polymerization	- Sol-gel - Coacervation

1.2.1 Macro-encapsulation

Millimeter and centimeter-scale encapsulation constitute what is known as macro capsulation (Bessai *et al.* 2021). By increasing the surface area, encapsulation of the PCM results in an increase in the rate of heat transfer, which in turn results in an increase in the thermal conductivity, in addition to this, it keeps the PCM from coming into direct touch with the building material and the environment around it, which contributes to an improvement in the PCM's stability (Wang *et al.* 2022).

İllustrates the process of encapsulating the PCM, with the PCM serving as the heart of the structure and the container capsule serving as the shell (Arunachalam, 2019). However, the degree to which the material of the shell is compatibles with the material of the building and the PCM is a subject that still has to be investigated for the sake of further improvement (Rathore *et al.* 2019). In the case of macro encapsulation, the following two methods are often used. The different types of microencapsulation techniques are grouped together in the following (Table 1.6).

Table 1.6 Type of macro-encapsulation (Vemmer *et al.* 2013)

Macroencapsulation techniques	
Physical	Chemical
- Gelation - Co-extruction - Coacervation	- Molecular inclusion - Cross-linking

In the physical method, we have several techniques, including gelation and emulsion extruction, which are the most widely used (Ayyaril *et al.* 2023).

- Gelation technique was created by Calvo and a colleague in 1997, In this technique (Figure 1. 3), polymer and polyanion solutions are combined to create nanoparticles and This method provides a straightforward and gentle approach to the synthesis of aqueous-based substances (Pan *et al.* 2002).

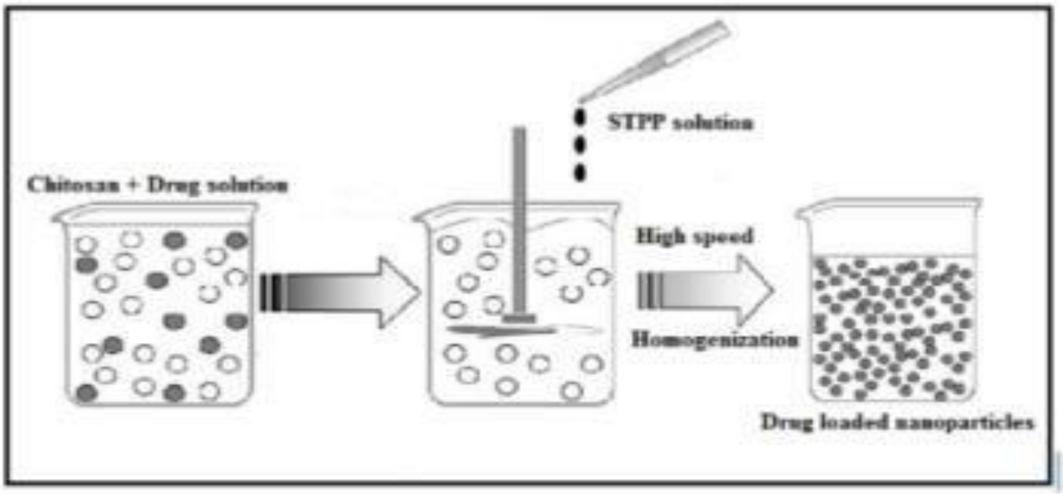


Figure 1.3 Schematic representation of the ionotropic gelation Method (Thuan *et al.* 2021)

- Co-extrusion is a process in which a dual fluid stream made up of liquid active ingredient and wall materials is poured into concentric tubes, particles are then generated as a result of the vibration that occurs as a result of this process (Ghosh, 2006) (Figure 1.4). The shell hardened through chemical cross-linking, evaporation of the solvent, or chilling and various types of extrusion nozzles were devised to optimize the process.

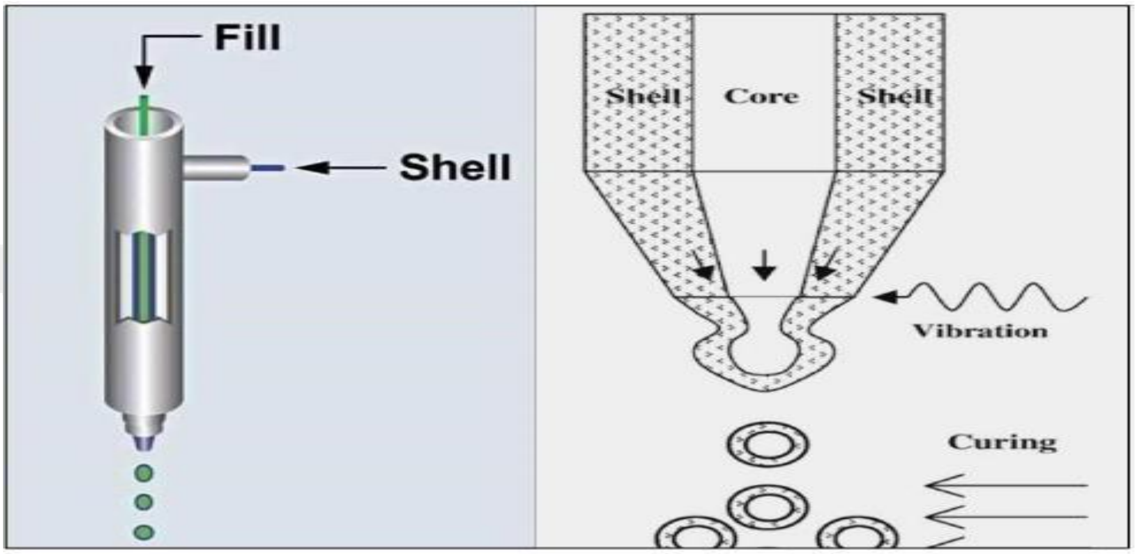


Figure 1.4 The Co-extrusion process (El-Kader *et al.* 2020)

Regarding the chemical method of macro encapsulation, two methods are often used for their synthesis and they are also described in the following section (Bharadwaz *et al.* 2020).

- Molecular inclusion, the association of an active component within a cavity-based substance is known as a molecular inclusion and Cyclodextrin is the most well-known example of this, these methods are more expensive, they are utilized far less frequently and Cyclodextrin contain a lipophilic inner pocket that is around 5-8 angstroms in size (Nedovic *et al.* 2011). If an active molecule has the correct size, it is possible for it to be reversibly entrapped within the cyclodextrin while being in an aqueous environment and the loading capacity of the ring is, however, hindered by the relatively tiny size of the hole that forms it and the shemas of the molecular inclusion method are presented (Figure 1.5) (Bachir *et al.* 2023).

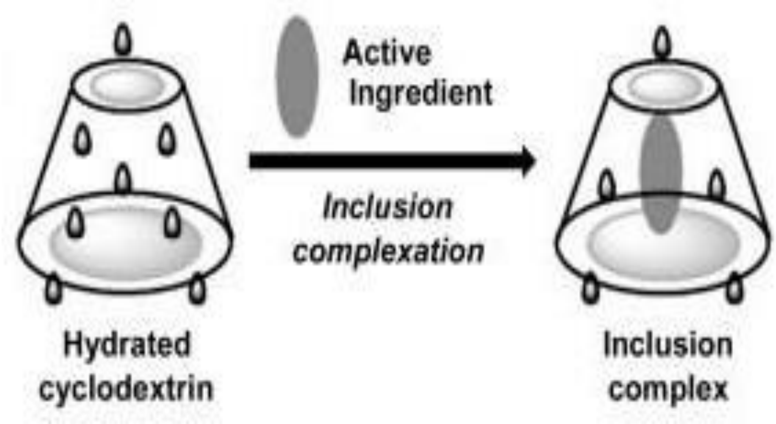


Figure 1.5 Molecular inclusion method (Bhushani *et al.* 2017)

- Cross-linking

Crosslinking the function group of the coating with the function group of the crosslinking agent is one way that can be used to accomplish encapsulation through the crosslinking method (Madl *et al.*, 2016). In the field of polymer science, the cross-linking agent refers to a material that, in the process of promoting or regulating the formation of intermolecular covalent bonds between polymer chains, links those chains together to form a more rigid structure (Al-Shannaq *et al.* 2015).

Illustrates how calcium ions are distributed throughout the polymer after being extracted from sodium algin (Figure 1.6). The shell starts to become more rigid and eventually turns into a microcapsule as a result of cross-linking that occurs between the carboxyl group and calcium ions.

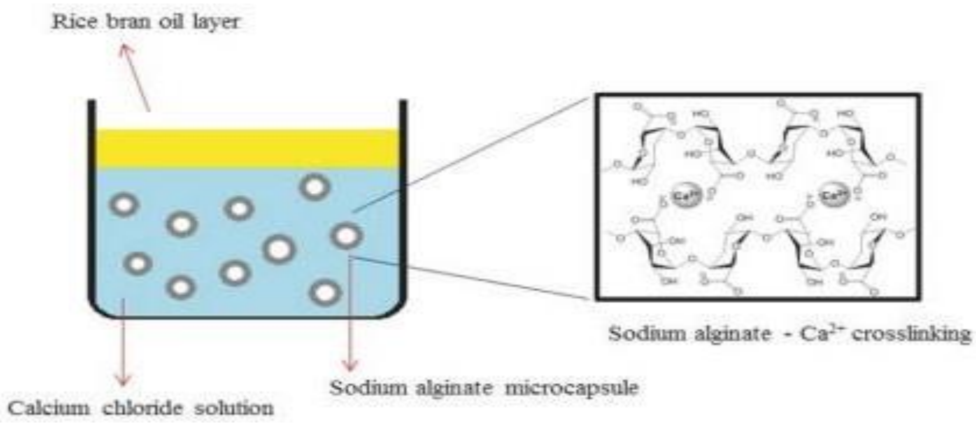


Figure 1.6 Sodium alginate crosslinking (Chaemsawang *et al.* 2018)

1.2.2 Micro-encapsulation

The microencapsulation of Phase Change Material (PCM) involves encapsulating minuscule particles or droplets of PCM in a protective shell or coating, this method utilized a variety of shell materials and encapsulation techniques to create microcapsules that enhance the management, stability, and efficacy of PCM (Tyagi *et al.* 2011).

Microcapsules, typically between 1 and 1 millimeter in diameter, are created through the process of microencapsulation, which employs a film-forming substance to enclose a solid or liquid (Augustin *et al.* 2001). According to the mechanism of synthesis, microencapsulation methods can be divided into three categories physical, chemical, and physical-chemical methods (Alva *et al.* 2017). There are several techniques for synthesizing PCMs in the micro-encapsulation process, including physical, chemical and physico-chemical methods. However, each technique has several other sub-methods to aid synthesis (Table 1.7).

Table 1.7 The classification of microencapsulation methods for PCMs (Peng *et al.* 2020)

Micro-encapsulation techniques		
Physical	Chemical	Physical-chemical
- Spray dyring - Solvant evaporation	- İn situ polymerization - İnterfacial polymerization - Suspension polymerization - Emulsion polymerization	- Coacervation - Sol-gel

The production of microcapsule shells in physical techniques solely requires physical processes such as spray drying, dehydration, and adhesion the two physical methods that are most widely employed for encapsulating PCMs are spray-drying and solvent evaporation (Huang *et al.* 2019).

- Spray dyring

The method of encapsulation known as spray drying is the one that is employed the most frequently and since the late 1950s, the food industry has been making use of this approach, which makes it one of the oldest methods of encapsulation (Mahdavi *et al.* 2014). This method has a number of benefits, including cheap operational costs, excellent quality capsules with a decent production, quick solubility of the capsules, a small size, capsules with high stability, and continuous operation (I Ré, 1998). The processes that make up the spray drying process are as follows firstly Atomizing the fluid feed in order to break it up into smaller droplets secondly evaporating the drying medium, third allowing the feed and spray to come into touch with one another and dry, and finally isolating the product from the air in the surrounding environment. The qualities of the finished product are directly influenced by each of these stages, as well as the operational factors governing how they are carried out. (Figure 1.7).

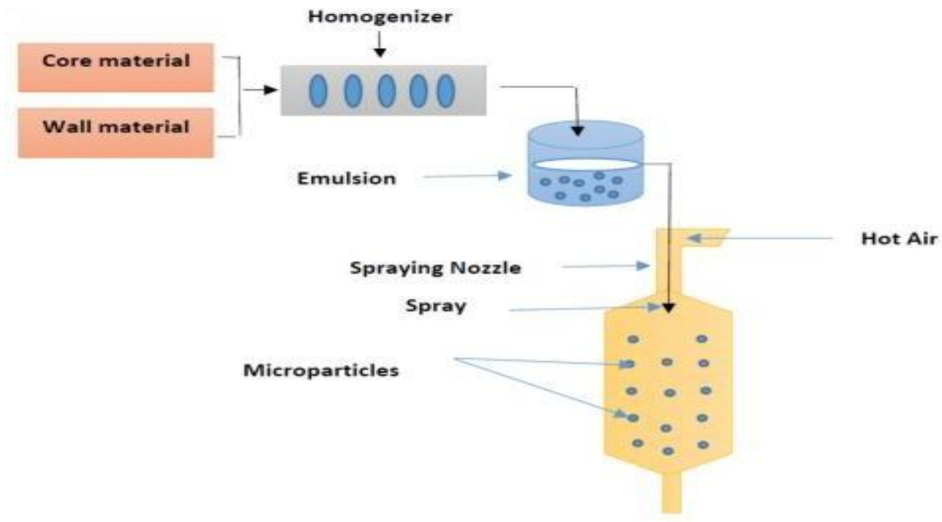


Figure 1. 7 Schematic representation of the encapsulation process by spray-drying (Mohammed *et al.* 2020)

- Solvant evaporation

Techniques for the evaporation of solvents have recently emerged as a more practical alternative to existing approaches, with this process, controlled particle sizes in the range of nanometers to micrometers may be accomplished (Tiwari *et al.* 2011). To accomplish good encapsulation efficiency and a low residual solvent content, however, requires a careful selection of encapsulation materials and a variety of conditions. (Carvalho *et al.* 2016). This method consists of four major steps such as dissolving the drug in an organic solvent, emulsifying the organic phase, extracting the solvent from the phase and finally recovering and drying the residual solvent (Figure 1.8).

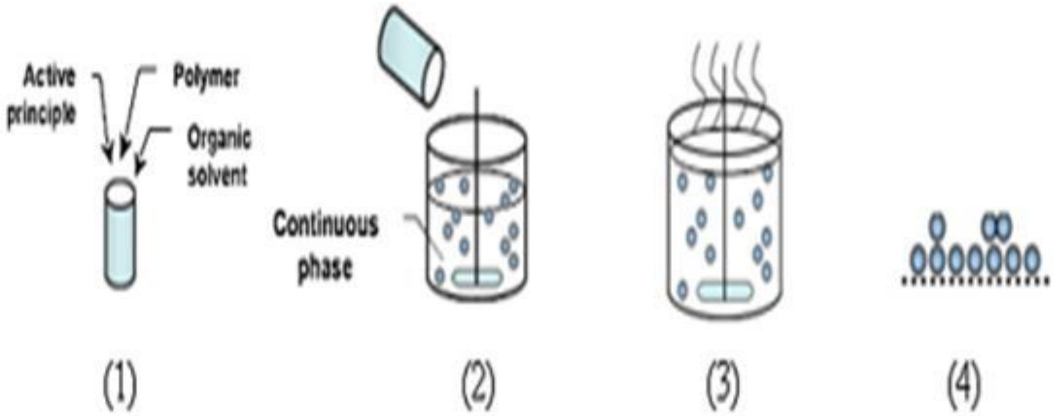


Figure 1.8 Basic steps of microencapsulation by solvent evaporation (Li *et al.* 2007)

The production of particles at the micrometer scale is also aided by chemical techniques which are considered to be efficient and important in the microencapsulation process and they are described as follows (Nandiyanto *et al.* 2011).

- Suspension polymerization

In order to generate microcapsules with a polymer that is based on PCM, one method that may be utilized is known as the suspension ploymerization procedure, In addition to this, the monomer must be dispersed in the form of a liquid droplet in a medium that has been stabilized Figure 1.9. (Sánchez *et al.* 2008).

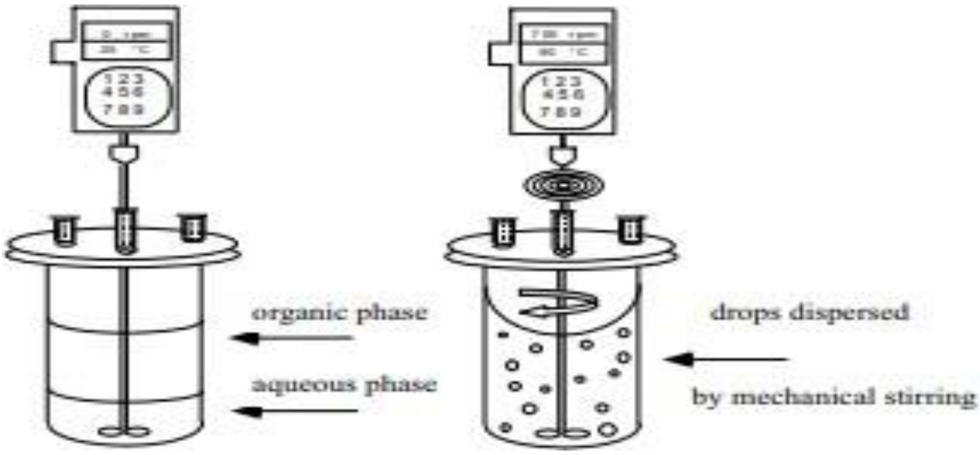


Figure 1.9 Schematic representation of suspension polymerization (Morejón *et al.* 2005)

- İnterfacial polymerization

Interfacial polymerization is a method that may be used to create polymers by combining monomers or pre-polymers that are dissolved in different phases that cannot mix, such as liquid-liquid or liquid-gas interfaces (Sivanathan *et al.* 2020). This approach is used to create polymers by combining monomers or pre-polymers that are dissolved in separate phases, at the point where the two different phases meet, this action causes the creation of a polymeric film at the interface (Pleşa *et al.* 2016).

In the process of interfacial polymerization (Figure 1.10), two reactive monomers are dissolved separately in the oil phase and the aqueous phase. Subsequently, polymerization takes place at the oil-water interface as a result of the action of an initiator.

Figure 1.10 Schematic representation of interfacial polymerization (X. Q. Cheng *et al.* 2014)

1.2.3 Nano-encapsulation

The term nanocapsulation describes a technique of enclosing substances at the nanoscale scale (Pereira *et al.* 2018). Nanocapsules have several applications outside of the medical and cosmetics sectors (Elkalla *et al.* 2023). The most important techniques for synthesizing nano-capsule phase-change materials are chemical techniques, which include emulsion polymerization, mini emulsion polymerization, in situ polymerization and interfacial polymerization (Table 1.8).Physico-chemical method (sol-gel) and a physico-mechanical method (electrohydrodynamic encapsulation)

Table 1.8 Nanoencapsulation techniques appropriate for PCMs (Alehosseini *et al.* 2020)

Nanoencapsulation techniques appropriate for PCMs		
Chemical techniques	Physico-chemical techniques	Physico-mechanical techniques
- Emulsion polymerization - Mini-emulsion polymerization - İn-situ polymerization - İnterfacial polymerization	- Sol-gel	- Electrohydrodynamic encapsulation

Chemical methods have successfully shown a major interest in the synthesis of phase change materials, emulsion and Mini-emulsion polymerization techniques are often used and are described as follows (Alehosseini *et al.* 2020).

- Emulsion polymerization

Emulsion polymerization is a one-of-a-kind chemical method that is utilized extensively in the production of aqueous resins with a wide range of colloidal and physicochemical characteristics (Chern, 2006). This technique of heterogeneous free radical polymerization requires the use of an oil-in-water emulsifie to emulsify the comparatively hydrophobic monomer in water (Kedzior *et al.* 2021).

Figure 1.11a depicts the two basic nucleation mechanisms involved in the emulsion polymerization process, micellar nucleation and homogeneous nucleation. (Figure 1.11b) shows the formation of polymer particles in the presence of monomer droplets, whereas (Figure 1.11c) consist the growth of polymer particles in the absence of monomer droplets. Both figures are included in the same figure. The four basic kinetic phases involved in the emulsion polymerization process are initiation, propagation, termination, and chain transfer. A detailed discussion of emulsion polymerization can be found elsewhere..

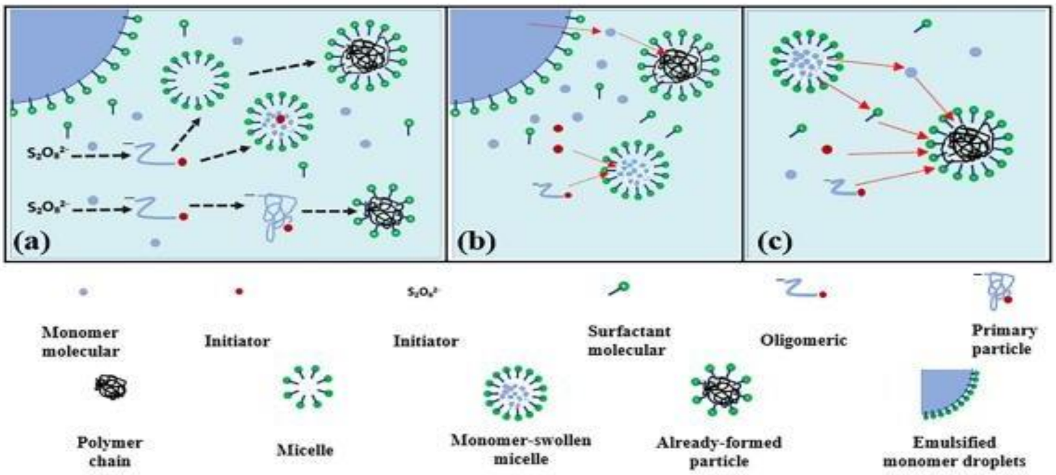


Figure 1.11 Process of emulsion polymerization: (a) Interval I, (b) Interval II, (c) Interval III (Zhang *et al.* 2021)

- Mini emulsion polymerization

Although emulsion and Mini-emulsion polymerizations are quite similar in many ways, there are significant differences in the processes of particle nucleation and monomer transport (Landfester *et al.* 2003).

Mini-emulsion polymerization is a type of polymerization that happens in each of the stabilized monomer nano-droplets, this sort of polymerization is also known as nano-droplet polymerization**,** the Ugelstad and colleagues were the first to describe the production of polystyrene polymer particles from monomer droplets with a size that was smaller than 0.7 micrometers (Mirzataheria *et al.* 2014).

The mixture first passes into the macro-emulsion state (Figure 1.12), after sonication the mixture passes into the Mini-emulsion process and the polymer-based molecule is formed.

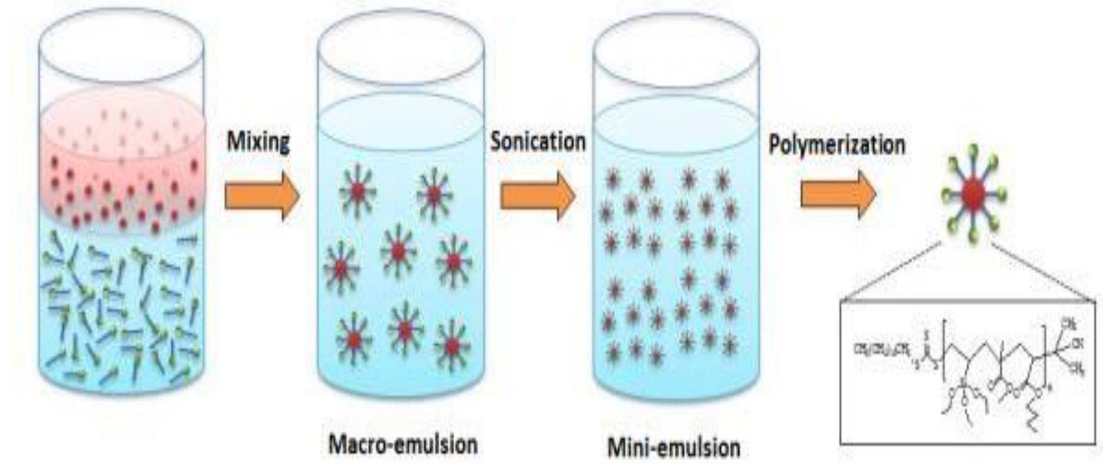


Figure 1.12 Representative image showing the preparation of copolymers via raft Mini-emulsion polymerization (Yilmaz *et al.* 2017)

The physical methods used to synthesize nano-encapsulations are described in the following section**.**

- Sol-gel

The method of immobilizing and protecting materials by enclosing them in a solid matrix produced by the sol-gel process is referred to as sol-gel encapsulation (Lin *et al.* 1997). The term phase change material (PCM) refers to substances that can store and release thermal energy via a phase transition, often from solid to liquid and vice versa, this transition allows the substance to store and release energy in a controlled manner (Pasupathy *et al.* 2008).

The sol-gel encapsulation technique has a lot of advantages to offer, these advantages include precise control over particle size and distribution, better stability and durability, and protection of the PCM from external forces (Ghasemi *et al.* 2022). Here are the operating principles of the sol-gel method for nano-encapsulation of phase-change materials (Figure 1.13).

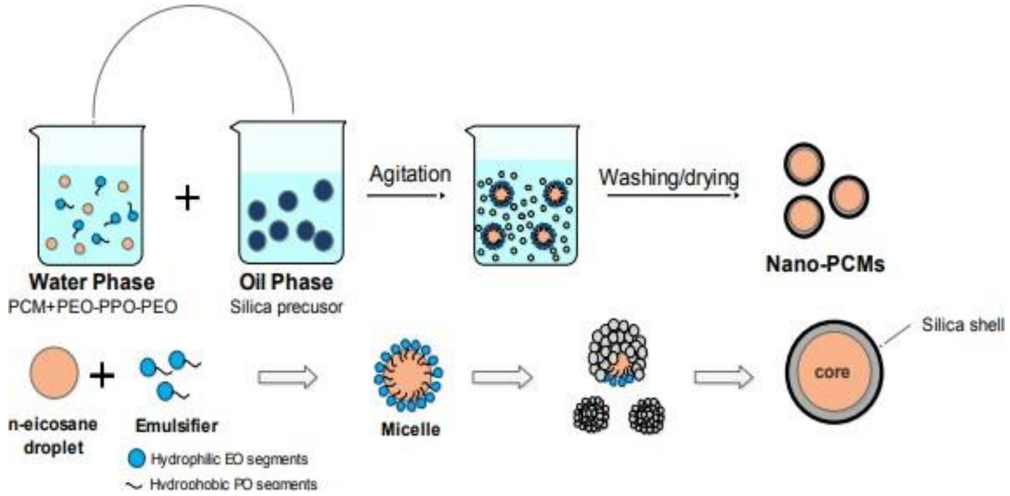


Figure 1.13 Schematic diagram of nano-encapsulated n-eicosane PCM with silica shell via sol-gel process (Route, 2017)

- Electrohydrodynamic encapsulation

Electrodynamic atomization is a simple and one-step physical process that has become a viable method for the fabrication of nanofibers and nanoparticles (Rostami *et al.* 2019). This technology has recently been shown to be beneficial for the encapsulation of a variety of diverse chemicals, such as biomedical drugs, functional food components, and phase change materials, with substantial yields and flexibility in design, resulting in the formation of micro, submicro and nano-sized structures (Chalco-Sandoval *et al.* 2015). Electrospray was capable of being utilized in a variety of settings (Figure 1.14).

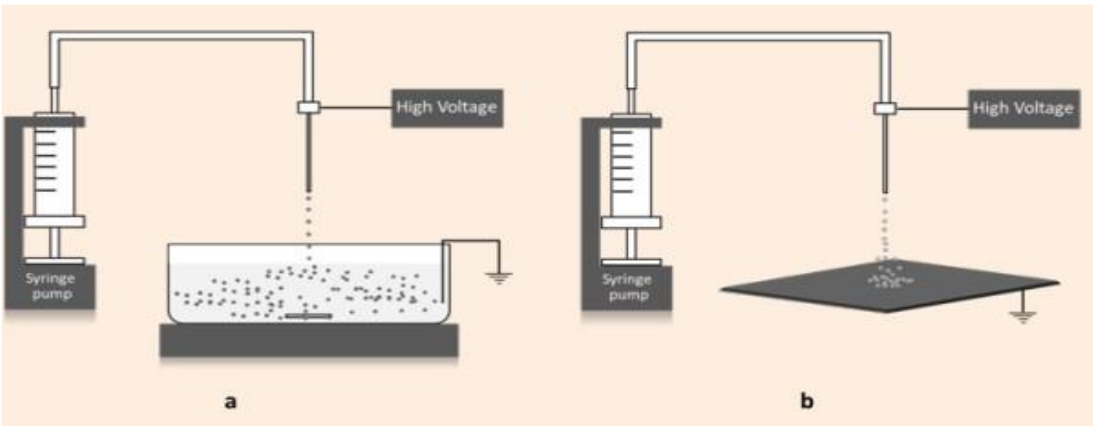


Figure 1.14 Schematic description of simple electrospray system using two types or collectors a Cross linking bath b Plate (Nikoo Mehregan *et al.* 2015)

1.3 Encapsulation of PCM with Silicat

The natural world is rich in silicates, a class of minerals and chemical substances (Eroglu *et al.* 2017). Their lattice structure of silicon and oxygen atoms with various cation associations sets them apart (Wanninger *et al.* 2015). One category of inorganic PCMs that has recently attracted interest due to its distinctive features and prospective uses is silicate-based PCMs (Hassabo *et al.* 2015). Compared to more common organic building components, silica offers several benefits (Knickerbocker *et al.* 2008). It is the appropriate solid matrix for adding other functions since it is inert, transparent, stable, biocompatible, and non-toxic (Sharma *et al.* 2011).

Silicate encapsulation is crucial due of the benefits it provides (Yang *et al.* 2013). The silicates can be shielded from degrading factors including moisture, oxidation, and unfavorable chemical interactions by being encapsulated (García *et al.* 2011). In addition, the silicates encapsulation allows for the modification of attributes including solubility, thermal stability, and reactivity (He *et al.* 2014). Because of its adaptability, new uses can be found in many different areas (He *et al.* 2015). (Figure 1.15) below shows silica encapsulation and its reaction mechanism.

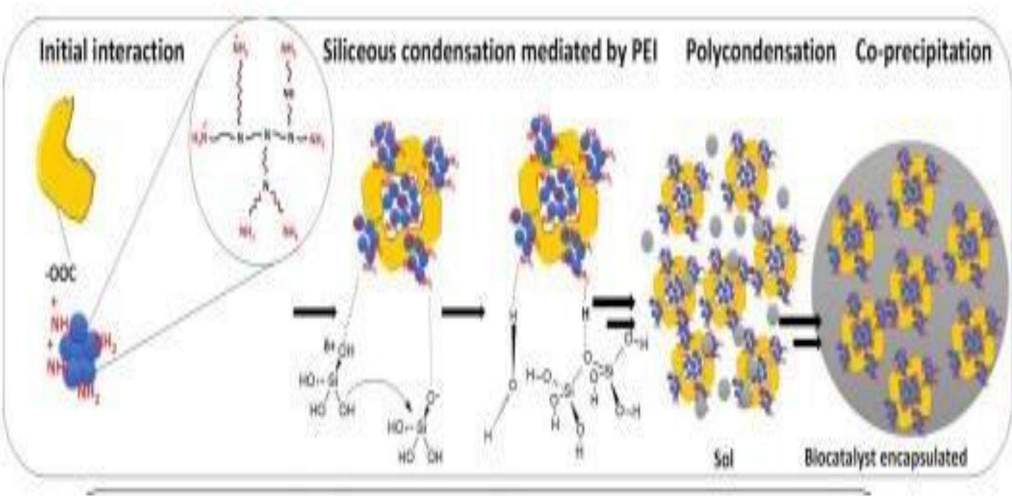


Figure 1.15 Mechanism of silica encapsulation (Escobar *et al.* 2018)

There are several uses for encapsulated silicates, examples of substrates that can benefit from protective coatings include metals and polymers, these coatings, which are based on encapsulated silicates, increase the durability and scratch resistance of the substrates (Q. Zhu *et al.* 2022). Composite materials often make use of them to improve mechanical or thermal qualities (Nikzad *et al.* 2011). Controlling the release of active catalysts improves their effectiveness and stability, and this is where encapsulated silicates come in (Misra *et al.* 2013). They are being researched for potential use in the sustained release of medicinal and cosmetic ingrédients (Salas *et al.* 2023)**.** Once encapsulated, silicates can be added to construction materials for better insulation, strength, or fire resistance (Z. Huang *et al.* 2021).

1.3.1 Production method of silicat nanoparticles

The use of silica nanoparticles is widespread and prevalent in a variety of technical and biological applications such as biosensors, catalysis and semiconductor polishing (Ghosh Chaudhuri *et al.* 2012). There have been several discussions on the primary methods that may be used to synthesize silica nanoparticles including the precipitation method and the micro-emulsion technique however, the stober process is of particular importance to us in this cas (Drummond *et al.* 2014).An efficient approach for the manufacture of monodispersed silica particles was initially found in 1968 (Bogush *et al.* 1988).

This method comprises the hydrolysis of tetra alkyl silicates in a combination of alcohol and water with ammonia serving as a catalyst. Since that time, the Stober technique has been thoroughly researched, and as a result, particles with sizes ranging from tens of nanometers to a few microns have been produced (X.-D. Wang *et al.* 2010). With the Stober method, it is possible to acquire exceptional control over the synthesis of both silica and non-silica particles. By modifying the composition of the reaction, it is possible to synthesize mesoporous silica nanoparticles of homogenous size using a Stober-like strategy. Figure 1.16 shows stober process.

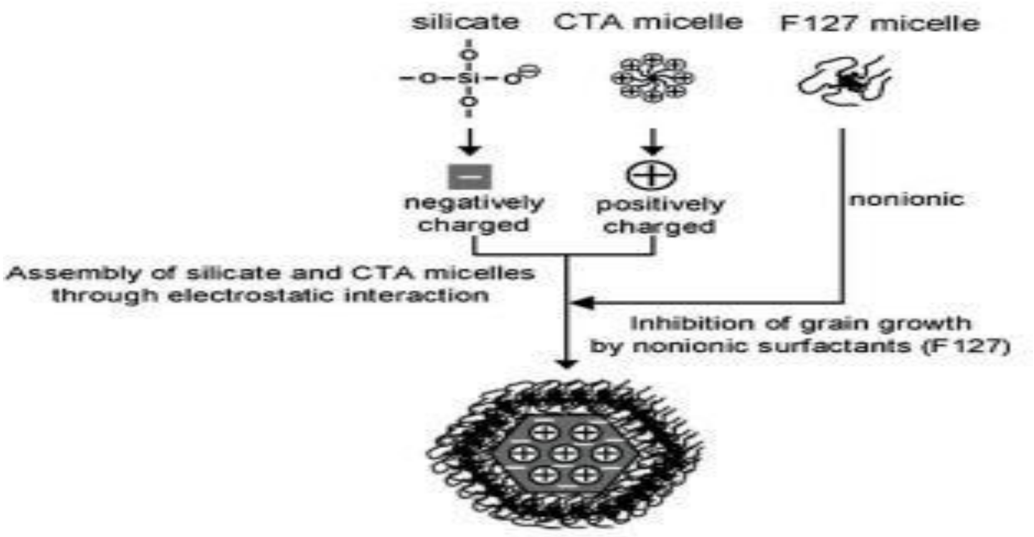


Figure 1.16 Stober process (Wu *et al.* 2013)

1.3.2 Properties of PCM with silicat

The use of any porous silica based thermal energy system is dependent on the characteristics of the porous silica (X. Huang *et al.* 2019). The temperature at which the phase transition occurs and the capacity of the material to store thermal energy are the two most essential thermal parameters for any phase change material (Pielichowski, 2014). Additionally, it is preferable to have a high specific heat and thermal conductivity since these characteristics boost the systems capacity to store sensible heat and its power density (Fernández *et al.* 2010) The different types of properties of silica phase-release materials (thermal, physical, chemical, and economic), their values and benefits are listed in Table 1.9.

Table 1.9 Desired properties of porous silica–based phase change materials (Mitran *et al.* 2021)

Type	Property	Value	Benefits
Thermal properties	Heat of fusion Specific heat Thermal conductivity Melting point	High High High	Increased energy storage density Increased power density, Determines operating temperature, lower temperature gradient
Physical properties	Volume change on transition Vapor pressure Crystallization rate Supercooling degree	Low Low High Low	Increases stability, minimizes leakage Decreases evaporative loss of material Decreases the hysteresis between charging and discharging
Chemical properties	Thermal & chemical stability Reactivity/corrosiveness Non-toxic, non-flammable, Non-explosive Wettability & surface tension	High Low High High	Increases life cycle Increases safety and decreases complexity Formation of shape stabilized materials with the silica Matrix and higher PCM loading
Economic properties	Cost Abundance Environmental impact	Low High Low	Improved economic efficiency and decreased risk

2. LITERATURE REVIEW

Various techniques for synthesizing silica nanoparticles have been the subject of published research. The following investigations are summarized as an example (Singla *et al.* 2022).

Miloudi *et al.* (2020) were studied Encapsulation of phase transition materials with nanostructured sodium carbonate and silicate added to alginate. The challenges of encapsulating phase change materials (PCMs) for thermal energy storage applications are discussed in this article. The authors propose a simple technique for manufacturing PCM encapsulation with both organic and inorganic shells. In the encapsulation procedure, a coaxial microfluidic system and ionic cross-linking are utilized. Using scanning electron microscopy, the compatibility and stability of the capsules with organic compounds are investigated, as well as their morphology. Athermal transform infrared spectroscopy (ATIR-FTIR) is used to analyze the capsules' chemical composition. The capsules are evaluated using thermogravimetric analysis, scanning electron microscopy, and elemental distribution analysis (EDX). This research provides valuable insights into the production and characterization of PCM encapsulation with both organic and inorganic coatings, offering potential advancements in applications for thermal energy storage. By effectively encapsulating PCMs, the difficulties of leakage, flammability, and environmental reactivity can be mitigated, resulting in enhanced performance and safety in thermal energy storage systems.

Yang *et al.* (2021) were examined phase change material-based thermal energy storage. This study demonstrates the potential of phase-change materials (PCMs) with high latent heat for applications involving thermal energy. However, their limited conductivity limits their power density and overall storage efficiency. To overcome these obstacles, the study stresses the importance of comprehension of multi-physics phase change phenomena, optimization of device design, incorporation of high-capacity PCMs, and the development of enhanced transport and thermodynamic characteristics. It is adequately described the fundamentals of heat transfer in PCMs, their properties, and the significance of their production. In addition, the article discusses the fundamentals, integration, and potential future advantages of PCMs. The results demonstrate the importance of advancing the understanding and application of PCMs to improve thermal energy storage systems. By addressing these factors, it is possible to harness the maximum potential of PCMs and optimize their use in various thermal energy applications, resulting in enhanced performance and efficiency.

Marske *et al.* (2022) were evaluted The effect of surfactants and organic polymers on the synthesis of monolithic shape-stabilized phase transition materials using the sol-gel method. Shape-stabilized phase-change materials (ss-PCMs) have the potential to store up to five times more thermal energy than PCM composite panels currently on the market. Their limited mechanical stability however poses a challenge. The focus of this study is the synthesis of ss-PCM with enhanced stability. This study investigates the formation of ss-PCM in situ and the effects of sodium dehydrocysulfate (SDS) and polyvinyl alcohol (PVA) on the properties of ss-PCM. By forming a silica scaffold through gelation and encapsulating PCM droplets with SDS in an O/W emulsion, excellent shape-stability (100%) and long-term performance (2000 heat cycles) are obtained, irrespective of the amount of porogen. Nevertheless, the dehydrating process fragments the silica, resulting in an increase in macropore diameter and a decrease in compressive strength (1.2 MPa) and thermal conductivity (37 W/mK). Excessive SDS concentrations have a negative impact on emulsion stability, diminishing compressive strength (1.02 MPa) by 63% and thermal conductivity (0.37 W/mK) by 25% (10 °C), respectively, during drying.

Ibrahim *et al.* (2010) studied the preparation of spherical silica nanoparticles. Using ethanol and ammonia as a catalyst, silica nanoparticles were produced by hydrolyzing and condensing TEOS. Moreover, they looked into how different concentrations of TEOS and NH3 affected the size of the silica nanoparticles and found that the size of the silica particles increased linearly as the rate of hydrolysis and condensation increased, which in turn affected the solubility of the intermediate [Si (OC2H5)4-X(OH)X] and, in turn, the supersaturation for the nucleating process. Finally, they use a technique called transmission electron microscope or TEM to see what is the effect of the change of the surface on the size of silica nanoparticle it has been**.** Noticed that during the modification of the surface the solution becomes hydrophobic and gives a good dispersion of silica nanoparticles.

Nandanwar *et al.* (2015) performed work on the production and characterisation of silica nanoparticles using an innovative Sol-Gel technique. To begin, tetraethylorthosilicate (TEOS), polyethylene glycol (PEG) 5%, and 0.001 N hydrochloric acid were used in a chemical synthesis to create silica nanoparticles. The nano-silica gel was made using the sol-gel technique. Hydrolysis and condensation processes on TEOS, a silica precursor, make up this technique. After determining the best conditions for silica nanoparticle production and processing, For the purpose of characterizing the particles that we got, we made use of x-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). It appeared from the data that the Silica nanoparticles had produced properly. The produced samples undergo a phase transition from amorphous to -crystallite. The findings of the XRD analysis demonstrated that the manufactured silica nanoparticles have an amorphous structure. Furthermore, the SEM and TEM images demonstrated that the particles were consistently sized at around 34 nm. From an energy-efficiency standpoint, our research suggests that the mild sol-gel reaction technique is preferable. The capacity to regulate the nanometer-scale internal structure of items was another bonus of this method.

Rao *et al.* (2005) demonstrated an innovative approach to the production of silica nanoparticles. For the first time, ultrasonication by the sol-gel technique has been applied in a sequential way to generate monodisperse and uniform-size silica nanoparticles. Particles of silica were obtained through the hydrolysis of tetraethyl orthosilicate (TEOS) in an ethanol medium, and the effect of several reagents on particle size was thoroughly studied. Particles ranging in size from 20 nm to 460 nm were successfully produced. In solutions of ammonia (2.8-28 mol L1), ethanol (1-8 mol L1), water (3-14 mol L1), and TEOS (0.012-0.12 mol L1), Particle size was analyzed using scanning electron microscopy and transmission electron microscopy. Furthermore, the importance of temperature's impact on particle size was evaluated. The results of this investigation are in line with those obtained from earlier studies that made use of UV-vis spectrophotometry to investigate the manner in which silica particles exhibited electronic absorption.

Ferreira *et al.* (2009) , Synthesis and characterisation of magnetite nanoparticles with silica coating were reported. Magnetic nanoparticles covered with silica have been the subject of study that is both extensive and, in many respects, intensive. This is because magnetic nanoparticles have the potential to be applied in a number of different technological sectors, most notably biomedicine. The nanoscale magnetite sample was synthesized using reduction precipitation, and the nanomagnetite particles were coated using the sol-gel process, which is predicated on the hydrolysis of tetraethyl orthosilicate (TEOS). The materials generated at room temperature were characterized by powder X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy (spectra were taken at different wavelengths), saturation magnification tests, and 57Fe Mössbauer spectroscopy.

Van Der Voort *et al.* (2002) were evaluated A New Templated Ordered Structure with Combined Micro- and Mesopores and Internal Silica Nanocapsules. Materials scientists have been inspired to seek out novel catalysts supports by the growing awareness of the need to develop environmentally friendly manufacturing methods across all areas of chemistry. These innovative catalytic supports should allow the heterogenization of most catalytic processes, which will increase the efficiency and selectivity of synthesis while minimizing waste and by products. As numerous micellar template structures, such as M41S1, FSM-16, HMS, and MSU-x, have been developed, it is now necessary to create support materials, consisting of a composite matrix with integrated micro- and mesoporosity and a sufficient stability to withstand most industrial treatments. In this study, we detail the first-ever creation of a hexagonal material that contains microporous silica nanocapsules inside its enormous pore sizes and thick walls (4 nm). Both wall- and nanocapsule-derived micropores are present in these plugged hexagonal templated silicas (PHTS), and the ratio of open to encapsulated mesopores is adjustable. The overall pore volume is greater than 1 cm3 //g, and the micropore volume is rather large (up to 0.3 cm3/g). The resulting materials are very stable, withstanding high hydrothermal temperatures and mechanical pressures with little to no deformation.

Wibowo *et al.* (2014) were examined Sustained Release of Fipronil Insecticide in Vitro and in Vivo from Biocompatible Silica Nanocapsules. A pesticide delivery system that is biocompatible and capable of sustained release is of great benefit to the agriculture industry. This study employs oil-core silica-shell nanocapsules that are biocompatible in order to accomplish continuous fipronil release. Using a dual-template synthesis strategy that combines emulsion and biomimetic techniques, nanocapsules are manufactured in a safe and environmentally favorable manner. By dissolving fipronil into the oil core prior to forming the silica shell layer, encapsulation efficiencies of up to 73% are attained. Experiments in vitro demonstrate that altering the silica shell thickness (8 to 44 nm) affects the fipronil release rate over time. Notably, in vivo laboratory studies demonstrate effective control of insecticidal activity against subterranean termites by regulating shell thickness. These results demonstrate the effectiveness and controllability of an environmentally friendly system for sustained insecticide release, indicating the system's potential for extensive use in a variety of agricultural applications with minimal environmental impact.

Wibowo *et al.* (2016) were studied Interfacial engineering for silica nanocapsules. Interest in silica nanocapsules has surged due to its intriguing core-shell structure. Medications, fluorescent dyes, and magnetic nanoparticles are just a few examples of the kinds of functional components that might be encapsulated in the core area, opening up a world of possibilities for use in drug delivery, imaging, and sensing. The biocompatibility, chemical and physical stability, and adaptable surface chemistry of the silica shell, however, make it an excellent protective covering for the enclosed payload. Scientists have spent a lot of effort researching how to create silica nanocapsules with certain features in order to suit the needs of numerous businesses. This survey provides an in-depth analysis of recent developments in silica nanocapsule production techniques, with a focus on the role played by different interfacial engineering approaches. This paper begins by introducing the various approaches to silica nanocapsule synthesis, with an emphasis on surface engineering of core template nanomaterials. Nanoparticles, liquid drops, and gas bubbles are all instances of basic templates. To control the nucleation and growth of silica at the interface between two phases (solid-liquid, liquid-liquid, or gas-liquid), this method employs chemical or biomolecular agents. The paper then go on to talk about how silica nanocapsules can be functionalized on their outer surface. Encapsulating active molecules within various capsule systems, including pre-loading and post-loading methods, is also discussed, along with the successes and failures of each. Finally, the paper explores the use of silica nanocapsules in imaging, theranostics, and controlled release.

Graham *et al.* (2021) were described Formation Mechanism of Multipurpose Silica Nanocapsules. Using Pickering emulsion templates and HCl-catalyzed condensation of tetraethyl orthosilicate (TEOS), this study demonstrates the successful fabrication of nanoscale capsules with an all-silica shell and an aqueous interior. As stabilizers in the formation of Pickering emulsions, modified commercial silica nanoparticles were utilized. The interfacial activity of the emulsifiers had a significant effect on the final capsule products' characteristics. Using stable Pickering emulsion templates and interfacially active particles, stable sub-micrometer core-shell structures were created. The resultant capsules displayed a silica colloidosome structure, with modified silica nanoparticles firmly affixed to the outer interface and surrounded by a thin but sturdy inner silica shell. By modifying the composition of the emulsion phases, the capsule size could be altered. Due to their unique structure, these silicic acid capsules have the potential to serve as dependable nanocarriers for hydrophilic active compounds in applications spanning from energy storage to drug delivery.

Y. Zhu *et al.* (2018) were considered Novel metal coated nano encapsulated phase change materials with high thermal conductivity for thermal energy storage. This study describes the development of nano-encapsulated phase change materials (Nano PCMs) with enhanced thermal conductivity that are coated with silver. Using Tollen's solution, n-octadecane-filled silica nanocapsules were surface-activated with dopamine and plated electrolessly with silver. XRD, FT-IR, XPS, and electron microscopy were utilized to determine the chemical composition, crystallinity, and morphology of the Nano PCMs. Using DSC, TG, thermal cycle experiments, and laser flash technique, the phase change properties, thermal stability, and thermal conductivity were determined. The silver-coated Nano PCMs exhibited decreased latent energies based on mass while retaining stable phase transition properties. In addition, the addition of silver increased the apparent thermal conductivity from 0.264 to 1.34 W/mK by a significant margin. Due to their remarkable properties, these silver-coated Nano PCMs hold great promise for applications in thermal energy storage and thermo-regulation.

Hassanajili *et al.* (2016) were examined Fumed silica polyurethane nanocomposites, effect of silica concentration and its surface modification on rheology and mechanical properties. This investigation examined the impact of nano silica type and concentration on the microstructure, mechanical properties, and rheology of TPU nanocomposites. In TPU nanocomposites, 5%, 10%, and 15% weight percent of unmodified silica (Si-Un), octylsilane-treated silica (Si-OS), and polydimethylsiloxane-treated silica (Si-PDMS) were used, respectively. Utilized characterization techniques included SEM, TEM, infrared spectroscopy, and DSC. The treated nanosilica improved dispersion, decreased particulate aggregation, and enhanced the interaction between the hard and soft phases of thermoplastic polyurethane (TPU), resulting in enhanced thermal stability and mechanical properties. Dynamic rheological analysis demonstrated that nanocomposites with treated silica possess superior viscoelastic properties. The incorporation of modified silica nanoparticles enhanced the TPU nanocomposites' mechanical properties and elasticity.

Imoisili *et al.* (2022) worked on the synthesis and characterization of silica nanoparticles by a new Sol-Gel method. The Sol-Gel method was utilized for the synthesis of silica nanoparticles. This procedure utilized tetraethylorthosilicate (TEOS), polyethylene glycol (PEG) at a concentration of 5%, and a solution of 0.001 N hydrochloric acid. To create nano-silica gel, the silica precursor TEOS was subjected to hydrolysis and condensation processes in the sol-gel technique. After optimizing silica nanoparticle manufacturing and purification conditions, the particles were characterized using x-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The analysis of the data revealed the effective synthesis of silica nanoparticles. There was evidence of a change in phase from amorphous to crystalline in the acquired samples. SEM and TEM pictures showed an average particle size of around 34 nm, and XRD examination confirmed the amorphous nature of the produced silica nanoparticles. According to our investigation, the moderate sol-gel reaction technique is preferable from an energy-efficiency standpoint. Moreover, this technique offers the benefit of controlling the internal structure of objects at the nanometer scale, which adds to its advantages.

Purcar *et al.* (2021) and other scientists were prepared silica nanoparticles and nanostructured solution of silica-gentamicin by microwave method in a basic medium with the antibiotic gentamicin sulfate and two silane precursors (tetraethylorthosilicate and octyltriethoxysilane). In this investigation, atomic force microscopy (AFM), transmission electron microscopy (TEM), and Fourier transform infrared spectroscopy (FTIR) were used to analyze the morphologies and structures of silica nanoparticles. These techniques provided significant insights into the properties of the investigated materials. The antimicrobial properties of silica nanoparticles and a nanostructured silica-gentamicin solution were compared. AFM analysis revealed morphological alterations in the nanostructured silica-gentamicin solution, including surface particle aggregation. FTIR analysis confirmed that gentamicin was successfully loaded into silica nanoparticles. Antibacterial experiments revealed that the nanostructured silica-gentamicin solution is highly effective against resistant strains. This silica-gentamicin solution has the potential to be used as a biochemical agent for preventing and treating microbial proliferation on a variety of surfaces, including ceramic and soil.

Tadanaga *et al.* (2013) studied the preparation of spherical silica nanoparticles. Silica nanoparticles were created through the hydrolysis and condensation of TEOS in ethanol and ammonia. Therefore, the proportional contributions of nucleation (phase shift or reorganization of the material) and process growth influence the size of silica nanoparticles. They also studied the impact of TEOS and NH3 concentrations on silica nanoparticle size and found that the solubility of the intermediate [Si (OC2H5)4-X(OH)X] and Consequently, the supersaturation for the nucleation process, increases with increasing concentrations of TEOS and ammonia. The size of modified silica nanoparticles is observed in a transmission electron microscope (TEM) study. The solution becomes hydrophobic upon surface modification, allowing for effective dispersion of silica nanoparticles.

Zulfiqar *et al.* (2016) described a technique for synthesizing silica nanoparticles from bentonite clay. A variety of thermal and acid treatments were applied to bentonite clay in order to lower the alumina concentration and raise the silica content. Sodium hydroxide solution (10 and 40% by weight) was applied to the resulting silica-rich clay to create silica sodium solutions (SSS). Three different concentrations of nitric acid (5 M, 10 M, and 15 M) were used to hydrolyze one kind of SSS with ethanol as a cosolvent, while three different volumes of ethanol were used to hydrolyze the second type of SSS (10 ml, 20 ml, and 30 ml). By varying the amounts of silica-rich clay, nitric acid, and ethanol, we were able to create silica particles with sizes spanning from the nanometer to the micrometer range. Particle size was found to be proportional to both silica-rich clay and HNO3 concentrations. As the concentration of ethanol in a 30 ml solution increased from 10 ml to 20 ml, nanometer- and micrometer-sized particles were produced. X-ray diffraction, scanning electron microscopy, and atomic absorption spectroscopy were used to learn more about clay, SSS, and nanoparticles, while inductively coupled plasma optical emission spectroscopy revealed similarities between the three.

Landau, (2008) were describe Synthesis and optimization of colloidal silica nanoparticles and their functionalization with methacrylic acid. Colloidal SiO2 nanoparticles were generated by catalyzing the hydrolysis and polycondensation of tetraethyl orthosilicate (TEOS) in an alcoholic solution with the right ratios of water, ammonia, and TEOS. The goal of this work was to create a unified model for predicting particle size as a function of experimental factors using a factorial design and multivariate statistical analysis. Under the correct conditions, including the right proportions of water, ammonia, and TEOS, this model predicted that silica nanoparticles with diameters between 17 and 333 nm could be formed. Only the NH3 molarity, the H2O molarity, and the influence from the molarity of TEOS and H2O interacting molarity were found to be statistically significant. Subsequently, 3(trimethoxysilyl)propyl methacrylate (TMSPM) was used to do in situ surface modification of the colloidal silica.

Jimenez *et al.* (2016) have investigated the fabrication of 20nm silica nanoparticles wrapped in three different photosensitizers which is based on porphyrin prepared by sol-gel method with the presence of cetyltrimethylammonium bromide with chemical formula $C_{19}H_{42}BrN$ and tetraorthosilicate with chemical formula TEOS as a silica source. The goal is to use photodynamic therapy to treat cancer cells. At 37°C in cell culture media, the colloidal stability caused by the PEG-silane chain grafted on the surface was shown. After being functionalized with silylated mannose squarate, the nanoparticles were employed in one- photon therapy on MCF-7breast cancer cells. After 5 hours, promising results were obtained.

Basu *et al.* (2015) have studied the toxicity of certain chemical elements on human life, namely uranium and thorium, which present toxicological effects. In this work, silica microspheres were used to decontaminate trace elements such as uranium and thorium from various underground and lake sources. The microspheres were separated from water samples using the ultra-filtration process. Using a sol-gel method and polymerization of polyvinylpyrrolidone emulsion, silica microspheres were created. The size of the microspheres was determined using dynamic light scattering, and the presence of the silicate structure was confirmed by measuring their attenuated total Reflectance. Recording of their infrared spectra with reduced total reflectance and Fourier transform. Using a scanning electron microscope, the surface morphologies of the produced silica microspheres were investigated. Uranium and thorium were added to Water samples taken from groundwater and lakes in doses ranging from 100 to 1000mg mL-1. The silica microspheres are very effective at pH 3 and have an asorption rate more than 99.9%, according to the experimental findings. The maximal absorption of uranium and thorium is 30 mg g-1 and 36 mg g-1, respectively, in the equilibrium sorption, which is governed by Langmuir isotherms. Before and after the purification operation, the major physico-chemical properties of the Water were observed. The experimental findings indicate that none of the measured parameters have changed significantly.

3. Materials and Method

3.1 In General

1. Water phase: 9.36mg of NH_4Cl is dissolved in 17.5mL of ultra-pure water and its pH is adjusted to 9 by adding 20 µL NH_4OH.

2. Organic phase: 10 mg of nonadecane is weighed and dissolved in 2.5 mL cyclohexane. 8 mL of tetraethyl orthosilicate (TEOS) is added to it and mixed thoroughly (or it can be mixed for a few seconds with the help of an ultrasonic probe (UP).

3. The organic phase is added to the water phase and emulsified with the help of UP (1 min, 30% power).

4. The reaction is continued for 24 hours on the magnetic mixer, in the oil bath, under the back cooler. Mixing speed 700-900 rpm.

5. Nanoparticles (NP) are precipitated and washed by centrifuge. It can be stored in water at room temperature or +40°C. The stock concentration should be more concentrated than 2mg/ML.

Since the particle size will be small, there is a possibility of non-collapse; therefore, if any, it can be precipitated by ultracentrifuge (30-40 thousand g). If there is no ultracentrifuge, the NP_S can be concentrated with the help of amicon or vivaspin (100K) or purified with the help of dialysis membrane (30-100 K). I normally use it directly without washing, the salt in it does not do any harm to me; however, if you are going to use it in the equipment, it may be corrosive.

All materials were purchased from Merck.

3.2 Chemical Used in The Experiments: Source and Purity

Table 3 1 Chemical used experiments

Chemical used solution	Formula	Vendor	Purity (%)
Ammonium chloride	NH_4Cl	Merck	99
Ammonium hydroxide	NH_4OH	Merck	25
Tetraethyl ortosilicate (TEOS)	$SiC_8H_{20}O_4$	Merck	99
Nonadecane	$C_{19}H_{40}$	Merck	99
Cyclohexane	C_6H_{12}	Merck	99

Equipment: Ultrasonic probe (Bandelin SONOPULS)
Magnetic stirrer
Magnetic rod
Reflux

3.3 Synthesis of Silicat Nanoparticles

Method: This method requires the emulsification of an oil phase in a water phase. These two different phases were prepared separately. First, water phase was prepared by dissolving 9,36 mg NH_4Cl in 17,5 mL milli Q water with 20 µL NH_4OH addition. The final pH should be around 9. Secondly, organic phase was prepared. 2.5 mL of cyclohexane solution with 10 mg nonadecane was added to 8 mL TEOS and mixed well. After all, the organic phase was added onto water phase and immediately emulsified by an ultrasonic probe (1 min, 30% power, 1 sec on and 1 sec off). The reaction continued at 40oC with 700-900 rpm stirring rate. After 24 h, the setting was disengaged and the solution was transported to a separation funnel to remove remaining cyclohexane. In separation funnel, the solution mixed well, and sat for 30 min without stopper before the valve was opened to obtain nanoparticle containing water phase. Nanoparticles were stocked at room temperature.

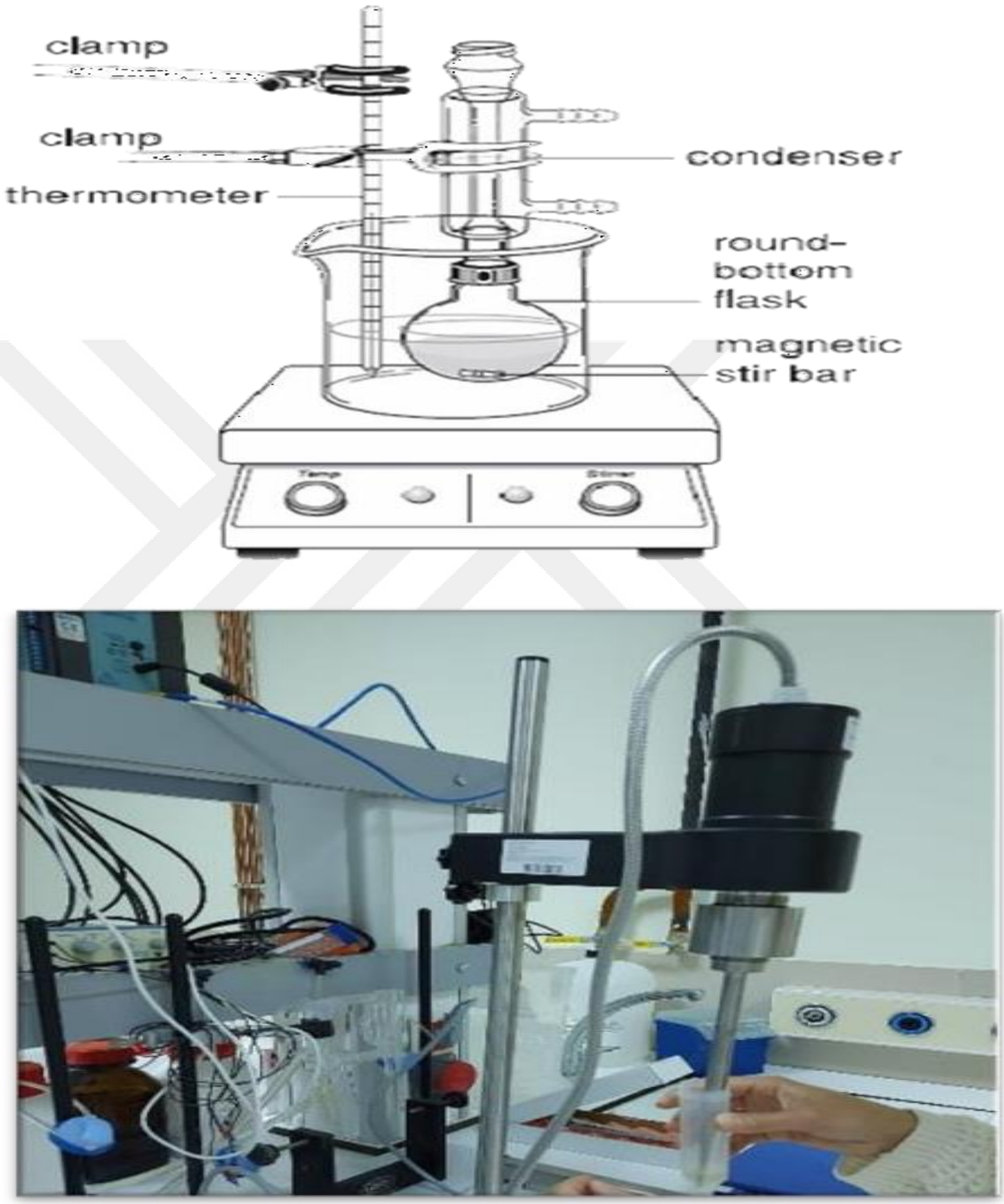


Figure 3 1 (1) Ultrasonicator device (2) Experimental setup for ultrasonication

4. RESULTS AND DISCUSION

4.1 SEM Analysis of Nanocapsules

SEM images were acquired at Çankırı Karatekin University Central Research Laboratory to determine the geometries of the perceived nano capsules. Figure 4.1 presents SEM images of empty nanocapsules without the presence of phase-changing substance (nonadecan), while Figure 4.2 displays SEM images of filled nanocapsules containing PCM. The anticipated geometry of the nanocapsules is spherical. The SEM images obtained confirm the spherical morphology of the nano capsules.

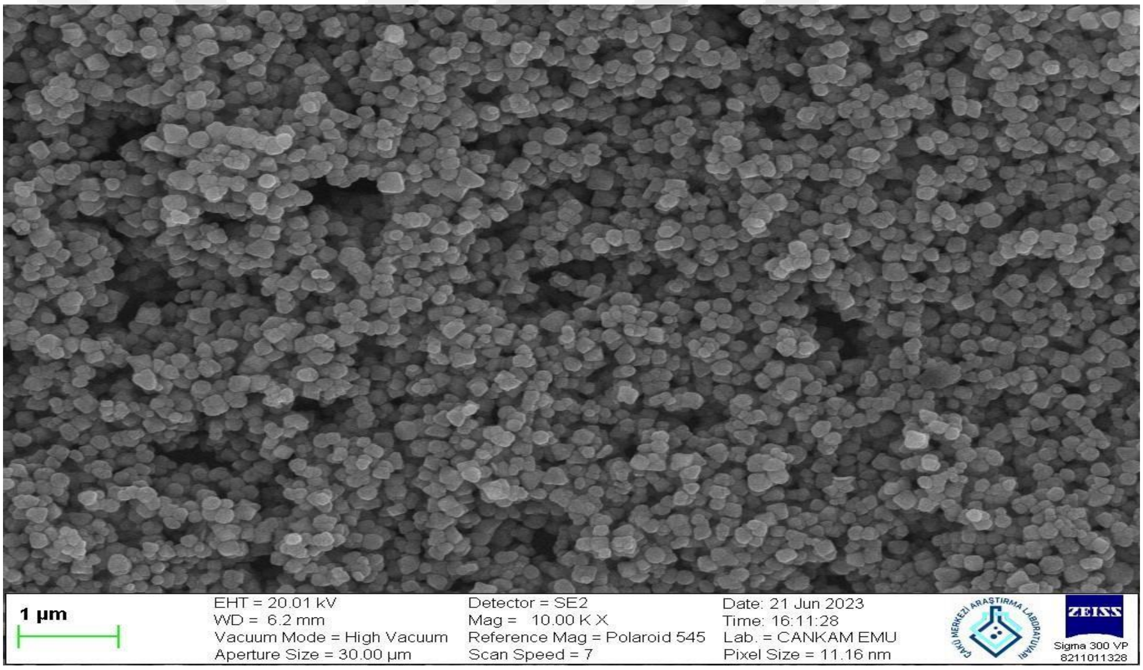


Figure 4 1 SEM images of empty nanocapsules without the presence of phase-change materials

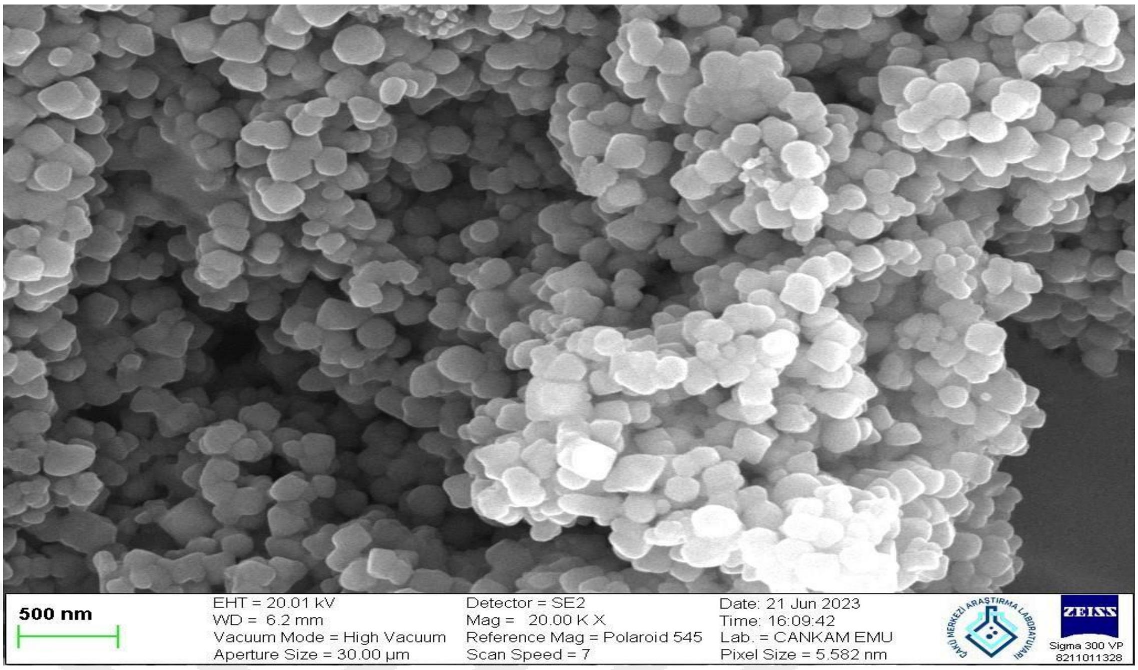


Figure 4 2 SEM images of empty silica nanocapsules

The images in Figure 4 reveal the accumulation of residual PCM outside the capsules after encapsulation. Washing the silica capsules with pure water at 40°C enables the removal of the non-encapsulated PCM.

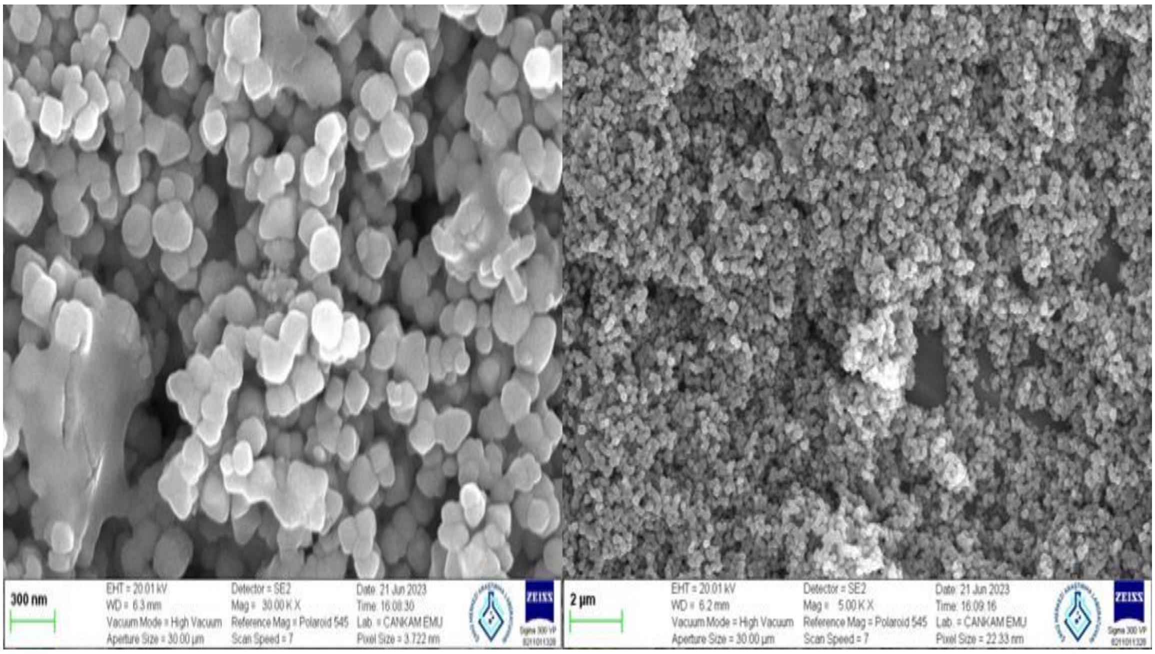
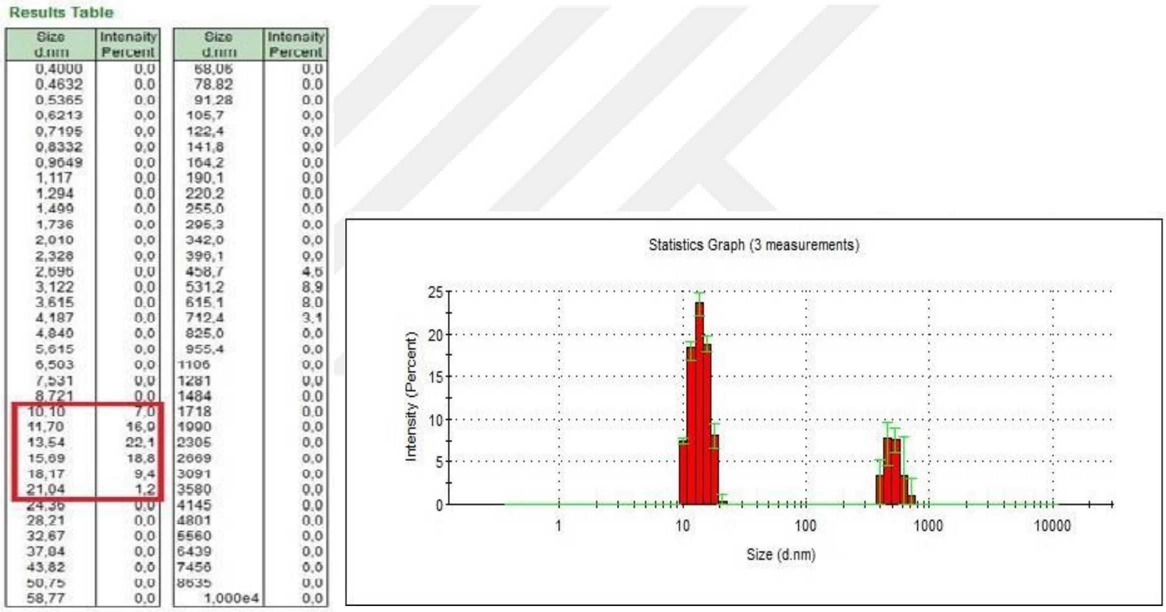


Figure 4 3 SEM images of nanocapsules containing PCM

4.2 Particle size

The particle diameters of the capsules were determined using the Malvern Zetasizer Nano ZS instrument for zeta potential and zeta size measurements at Çankırı Karatekin University Central Research Laboratory. The distribution of particle diameters is illustrated in Figure 5. Peak 1 is observed at 11.92 nm. When agglomerated particles are excluded from the distribution, the average particle diameter remains at 11.92 nm. Figure 4.3 (a) displays the percentage density of particle diameters. As evident from Figure 4.3 (b), particle diameters predominantly cluster around 10 nm.



Results Table

Size d.nm	Intensity Percent	Size d.nm	Intensity Percent
0,4000	0,0	68,06	0,0
0.4632	0.0	78.82	0.0
0,5365	0,0	91,28	0,0
0,6213	0,0	105,7	0,0
0,7195	0,0	122,4	0,0
0,8332	0,0	141,8	0,0
0,9649	0,0	164,2	0,0
1,117	0,0	190,1	0,0
1,294	0,0	220,2	0,0
1,499	0,0	255,0	0,0
1,736	0,0	295,3	0,0
2,010	0,0	342,0	0,0
2,328	0,0	396,1	0,0
2,696	0,0	458,7	4,5
3,122	0,0	531,2	8,9
3,615	0,0	615,1	8,0
4,187	0,0	712,4	3,1
4,840	0,0	825,0	0,0
5,615	0,0	955,4	0,0
6,503	0,0	1106	0,0
7,531	0,0	1281	0,0
8,721	0,0	1484	0,0
10,10	7,0	1718	0,0
11,70	16,9	1990	0,0
13,54	22,1	2305	0,0
15,69	18,8	2669	0,0
18,17	9,4	3091	0,0
21,04	1,2	3580	0,0
24,36	0,0	4145	0,0
28,21	0,0	4801	0,0
32,67	0,0	5560	0,0
37,84	0,0	6439	0,0
43,82	0,0	7456	0,0
50,75	0,0	8635	0,0
58,77	0,0	1,000e4	0,0

a

b

Figure 4 4 Particle Diameter Distributions of Nano Capsules

4.3 FTIR Analysis

FTIR Analysis of Stöber Silicas reveals characteristic peaks at 1100, 964, and 796 cm^-1, corresponding to symmetric stretching of Si-O-Si, stretching of Si-OH, and asymmetric stretching of Si-O-Si bonds, respectively [13].

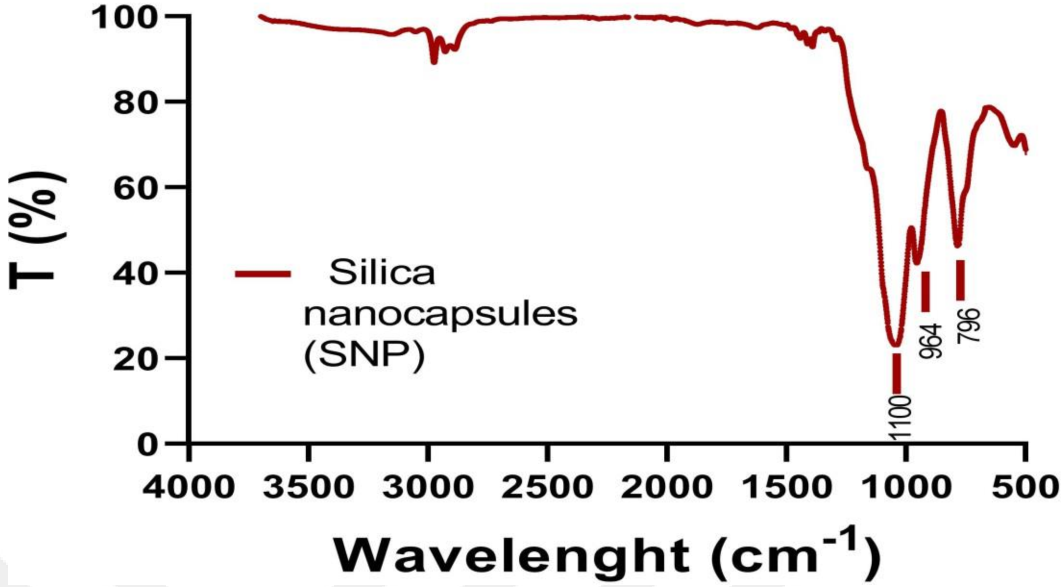


Figure 4 5 FTIR Analysis of Nano Capsules

4.4 DSC Analysis

The thermal properties of PCM (nonadecane) and PCM-containing nano capsules, such as phase transition temperatures (melting-crystallization temperatures) and latent heats, were determined using DSC. Figure 4.5 and Figure 4.5 display the DSC curves of pure nonadecane and nonadecane-containing nanocapsules during heating and cooling processes. The curves exhibit two endothermic and two exothermic peak points. The smaller peaks observed on the left side of the curves correspond to solid-solid phase transitions, while the main peaks represent solid-liquid or liquid-solid phase transitions. The melting and freezing temperatures were measured as 31.23 and 33,58 °C for nonadecane, and 29.13 and 33,9 °C for PCM-containing nanocapsules, respectively.

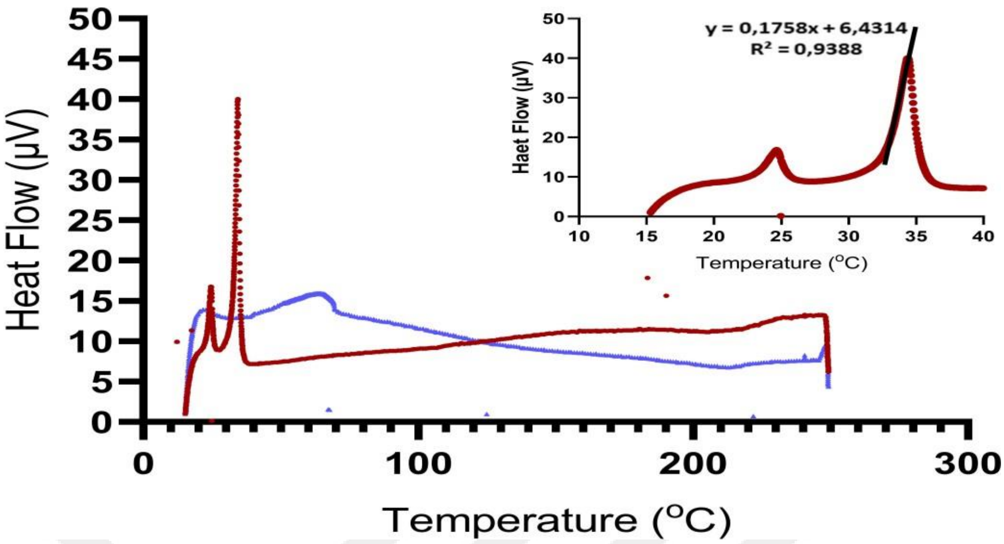


Figure 4 6 DSC Analysis of Nanocapsules

5. CONCLUSIONS AND RECOMMENDATION

In this study, silica nanoparticles were prepared using the emulsification method, and several parameters were examined through analysis and the following results were obtained.

— The shape of the nanocapsules, both with and without the PCM, is spherical, as determined using SEM.

— Due to the zeta potential, the diameter of the nanocapsules was successfully measured, yielding a value of 11.92 nm. However, based on clustering, the predominant diameter is approximately 10 nm.

— FTIR analyses of the Stöber method reveal that the peaks at 1100, 964, and 796 cm^-1 correspond to symmetric stretching of Si-O-Si, stretching of Si-OH, and asymmetric stretching of Si-O-Si bonds, respectively.

— The thermal properties of PCM (nonadecane) and PCM-containing nanocapsules, including transition temperatures for melting and crystallization, as well as latent heat, were measured using DSC.

The preparation of nanocapsules and the determination of several parameters, including shape, diameter, transition temperatures, fusion temperatures, and latent heat, are crucial for assessing the effectiveness of the nanocapsules.

Whether encapsulated or not, the shape of the nanocapsule remains unchanged (spherical).

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