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**CHARACTERIZATION OF THE CERAMIC RAW
MATERIAL PROPERTIES OF DONT CLAY
(FETHİYE, SW TÜRKİYE)**



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Oktaý Özdemir

27/09/2024

ABSTRACT
CHARACTERIZATION OF THE CERAMIC RAW MATERIAL
PROPERTIES OF DONT CLAY (FETHİYE, SW TÜRKİYE)

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Clays are very important materials for manufacturing ceramics. Although technological improvements play a very significant role in ceramic production, traditional methods are still used for pottery making. Clay material from the weathering profiles in the Dont plateau (Fethiye, Türkiye) has been used by the local population for pottery production for many years. A locally-known pottery recipe is the mixture of this brownish clay material, called Dont clay in this study, with a cream-colored clay material from the nearby vicinity in a 5:1 ratio. This mixture is called ceramic mud. Additionally, pottery decorations are made using a white-colored clay material applied with fingers. Although the structural features and mineral deposits of the region have been widely studied, the clay materials used for pottery production have not been examined comprehensively in terms of their raw material potential for industrial applications. Therefore, this study aims to characterize specifically the Dont clay along with the other clay materials used together to produce the ceramic mud in terms of mineralogical, geochemical and thermal properties. The sampling strategy was to collect the clay materials from the same locations where the local potter sources them. Parent rocks were petrographically examined under a polarizing microscope. All the clay materials as well as ceramic muds were mineralogically characterized by X-ray Diffraction and Fourier Transform Infrared Spectroscopy Analyses; geochemically (major oxide compositions and loss on ignition values) by X-ray Fluorescence Analysis. The phase changes and weight loss of the raw clay materials upon thermal loading were determined by Differential Thermal Analysis-Thermogravimetry. Additionally, XRD analysis was used to monitor the changes in the mineralogy of the clay materials by heating at 400° C, 800° C and 1000° C with 200° increments every 30 minutes. Lycian nappes and the Beydağları autochthon are the dominant geological features of the region providing gabbro, peridotite and limestone as parent rocks of the Dont clay. Dont clay samples (Dont clay sample and Esenköy sample) include almost identical mineralogy as talc, tremolite, quartz, plagioclase, calcite as non clay minerals with kaolinite and smectite as the major clay minerals. Esenköy sample has relatively more kaolinite (18%), higher Al₂O₃ (12.1%) and higher LOI value (10.15%) than Dont clay sample. Ceramic muds produced from the clay materials in 2021 and

2022 reveal very similar mineralogy with a minor difference in relatively higher abundances of talc bringing relatively higher MgO content. Talc increases the resistance of the bulk compositions to high temperatures ($\sim 950^{\circ}\text{C}$), however heating both Dont clay materials, additive and decorative as well as ceramic muds at 1000°C produces new crystal phase, cristobalite. A comparison of Dont clay materials and the ceramic muds with reference clays reveal that Dont clay materials are characterized by higher contents of silica and carbonate but lower contents of aluminum and clay minerals. This makes Dont clay samples unsuitable for structural clay ceramics or high-quality stoneware products.

Key words: Pottery, ceramics, talc, thermal, clay minerals



ÖZET
DONT KİLİNİN SERAMİK HAMMADDE ÖZELLİKLERİNİN
KARAKTERİZASYONU (FETHİYE, GB TÜRKİYE)

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Kil, seramik üretiminde çok önemli bir malzemedir. Teknolojik gelişmeler seramik üretiminde çok önemli bir rol oynamasına rağmen, çanak-çömlek yapımında hala geleneksel yöntemler kullanılmaktadır. Dont yaylasındaki (Fethiye, Türkiye) aşınma profillerinden elde edilen kil malzemesi, yerel halk tarafından uzun yıllardır çanak-çömlek üretiminde kullanılmaktadır. Yöresel olarak bilinen bir çanak-çömlek tarifi, bu çalışmada Dont kili olarak adlandırılan bu kahverengimsi kil malzemesinin, yakın çevreden gelen krem rengi bir kil malzemesiyle 5:1 oranında karıştırılmasıdır. Bu karışıma seramik çamuru adı verilir. Ayrıca, çanak-çömlek süslemeleri parmakla uygulanan beyaz renkli bir kil malzemesi kullanılarak yapılır. Bölgenin yapısal özellikleri ve mineral yatakları yaygın olarak incelenmiş olmasına rağmen, çanak-çömlek üretiminde kullanılan kil malzemeleri, endüstriyel uygulamalar için hammadde potansiyelleri açısından kapsamlı bir şekilde incelenmemiştir. Bu nedenle, bu çalışma, mineralojik, jeokimyasal ve termal özellikler açısından seramik çamurunu üretmek için birlikte kullanılan diğer kil malzemeleriyle birlikte Dont kilini özel olarak karakterize etmeyi amaçlamaktadır. Örneklem stratejisi, kil malzemelerini yerel çömlekçilerin kaynakladığı yerlerden toplamaktır. Ana kayalar polarize mikroskop altında petrografik olarak incelendi. Tüm kil malzemeleri ve seramik çamurları mineralojik olarak X-ışını Kırınımı ve Fourier Dönüşümlü Kızılötesi Spektroskopisi Analizleri ile; jeokimyasal olarak (ana oksit bileşimleri ve tutuşturma kaybı değerleri) X-ışını Floresan Analizi ile karakterize edildi. Ham kil malzemelerinin termal yükleme üzerine faz değişimleri ve ağırlık kaybı Diferansiyel Termal Analiz-Termogravimetri ile belirlendi. Ayrıca, her 30 dakikada bir 200° artışlarla 400° C, 800° C ve 1000° C'de ısıtılarak kil malzemelerinin mineralojisindeki değişimlerin izlenmesi için XRD analizi kullanıldı. Likya napları ve Beydağları otoktonu, Dont kilinin ana kayaları olarak gabro, peridodit ve kireçtaşı sağlayan bölgenin baskın jeolojik özellikleridir. Dont kil örnekleri (Dont kil örneği ve Esenköy örneği) ana kil mineralleri olarak kaolinit ve smesit ile birlikte kil dışı mineraller olarak talk, tremolit, kuvars, plajiyoklaz, kalsit olarak neredeyse aynı mineralojiyi içerir. Esenköy örneği, Dont kil

örneğine kıyasla nispeten daha fazla kaolinit (%18), daha yüksek Al_2O_3 (%12,1) ve daha yüksek LOI değerine (%10,15) sahiptir. 2021 ve 2022'de kil malzemelerden üretilen seramik çamurları, nispeten daha yüksek MgO içeriği getiren nispeten daha yüksek talk bolluğundaki küçük bir farkla çok benzer mineralojiyi ortaya koymaktadır. Talk, dökme bileşimlerin yüksek sıcaklıklara (~950 °C) direncini artırır, ancak hem Dont kil malzemelerini, katkı ve dekoratif hem de seramik çamurlarını 1000 °C'de ısıtmak yeni kristal faz olan kristobalit üretir. Dont kil malzemelerinin ve seramik çamurlarının referans killeriyle karşılaştırılması, Dont kil malzemelerinin daha yüksek silika ve karbonat içerikleri ancak daha düşük alüminyum ve kil mineralleri içerikleriyle karakterize edildiğini ortaya koymaktadır. Bu, Dont kil örneklerini yapısal kil seramikleri veya yüksek kaliteli seramik ürünleri için uygunsuz hale getirir.

Anahtar kelimeler: Çömlek, seramik, talk, termal, kil mineralleri



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LIST OF SYMBOLS

Å	Angstrom
°C	Degree Celsius
DTA-TG	Differential Thermal Analysis-Thermogravimetry
FTIR	Fourier transform infrared spectroscopy
mg	Milligram
TG	Thermogravimetry
XRD	X-ray Diffraction
XRF	X-ray Fluorescence

1. INTRODUCTION

1.1. Background of The Study

Clay is characterized as an extremely fine-grained material containing clay minerals, abundant in nature, rich in aluminium and silica and it develops plasticity when wet and hardened when fired (Velde, 1995). The existence of this natural substance has been known since the beginning of human civilization, and this has led it to play a significant role in every stage of human life (Konta, 1995; Bergaya and Lagaly, 2006). Its noticeable presence in different parts of the world and structural properties allow clay to have a broad range of uses and be utilized for diverse purposes (e.g., ceramics, building material, drilling mud, paints, coatings, polymers, films, health and wellness, etc.). One of the fundamental characteristics of clay is the resilience to various atmospheric conditions, depending on its structure and constituent elements. Moreover, its low cost and extensive availability in nature enable clay to be widely used as an industrial raw material in both Türkiye and the world (Ouahabi et al., 2014; Semiz and Çelik, 2020). Therefore, clay, based on its economic and functional advantages, has become a preferred material for various industries. Especially due to its thermal conductivity, clay is one of the most crucial raw materials in the ceramic industry (Vieira et al., 2008; Gualtieri et al., 2010). This thermal conductivity feature is of critical importance in the firing and temperature control processes of ceramics, and therefore, clay is regarded as an indispensable component in ceramic production facilities worldwide.

Türkiye has rich clay deposits in western Anatolia, which play an important role in the raw material and ceramic industry. Clay used in the ceramic industry is an important mining industry in Turkey, however, the increasing demand for industrial raw materials inevitably requires the development of new alternative clay sources (Agayev and Küçükuysal, 2018). This highlights the importance of effective and efficient use of resources for sustainable industrial development. In conclusion, a deeper understanding of the properties of clay and a more comprehensive study of the

potential uses of this natural material are essential for sustainable industrial development at the local and global levels.

According to Craig et al. (2013), the earliest ceramics and pottery were innovations of hunter-gatherers, dating back around 20,000 cal BP. From eastern Asia to Europe, the preparation and use of pottery transformed, revealing how social, economic, and climatic factors influenced its development. Established cultures in western Anatolia, from the Early Neolithic to present, have produced pottery with various textures, motifs, and paints, reflecting the characteristics of different regions and periods. For example, black-burnished pottery was common in the Early Bronze Age settlements in western Anatolia except the southwest (Fidan et al., 2015). From the past to the present, the raw materials used in pottery making have provided information about the lives, settlements, and even migration routes of cultures. Therefore, examining the raw materials used in ancient and contemporary ceramics, as well as determining their mineral and chemical constituents is quite important.

Settlers of Fethiye (SW Türkiye) continue the traditional pottery-making culture passed down through the generations using the soil from the Dont plateau. It is the region where archeological studies (Çaltılar Archeological Project) have been carried out. Knowing the historical background of the region on the traditional pottery making, the clay materials of the Dont plateau are selected for the present thesis study. The information regarding the clay materials in the Dont plateau is limited. Therefore, the major goal of this study is to carry a comprehensive characterization of the local clay materials of the Dont plateau in terms of mineralogical, geochemical, thermal and micromorphological properties. The innovative and original value of this work lies in its focus on an area that has never been explored before in the context of industrial raw materials and the comprehensive analysis of the clay materials found in this area. Although clay materials have been used in pottery in the region historically, there is a significant gap in the characterization of these materials in the geological literature. Therefore, this study fills this gap for the first time and provides an important basis for subsequent research and potential industrial applications. In the Dont plateau, local people prepare the pottery using traditional methods. Archaeological records indicate that similar pottery remains have been found in the Çaltılar Höyük (Fethiye). An interview was conducted with a local potter, Ms. Özen, regarding traditional pottery production. The sites where the raw materials for the pottery are sourced were visited.

Ms. Özen explained that the primary raw material is extracted from a pit about 40-50 cm deep in an area of the Dont plateau used for seasonal agriculture (Figure 1.1).



Figure 1.1. Clay pit in Dont plateau (This clay material referred to as Dont clay in this study (DC2022L02-M01))

The material is collected from the lower levels where there are fewer plant roots and pebbles. A soil profile typically consists of different layers known as horizons (Schoeneberger et al., 2012). At the top, the O horizon is found, which is the richest in organic matter. This layer consists of decomposing plant material such as leaves and branches, and it is the surface layer of the soil. Directly below the O horizon lies the A horizon, where plant roots and microorganisms are abundant. Further down, the B horizon is found. In this horizon, the amount of organic matter decreases, while clay, iron, and aluminum oxides accumulate. The B horizon is known for its higher mineral accumulation, particularly clay. The C horizon is the transition zone which includes the soil as well as the fragments of the parent rock (Schoeneberger et al., 2012). During the fieldwork, it was observed that this level corresponds to the B horizon in the soil profile, where clay accumulation is higher. Ms. Özen also mentioned that they use gray-beige material, collected from surfaces in the Dont Plateau where rocks are exposed and weathering is evident, as an additive to the primary raw material (Figure 1.2).



Figure 1.2. Weathering surface of the host rock in Dont plateau (clay material referred to as main additional material (DC2022L02-AM01).

After collecting both materials from the field, they leave them to air dry under normal conditions. Once dried, the materials are sifted through sieves with a 3mm mesh opening, breaking up clumps by hand or with a mallet (Figure 1.3).



Figure 1.3. Photographs showing the process of sieving A) the Dont clay; the clay aggregates are visible on the top of the sieve B) the additional material

The since fraction $< 3\text{mm}$ is then used for pottery making. After sieving, a mixture is prepared with 5 units of the primary Dont clay and 1 unit of the additive material. This mixture is kneaded with water until a plastic clay is obtained. The prepared clay is wrapped in plastic to prevent air exposure and left to rest for about an hour before being ready for pottery making. The clay is then shaped on a potter's wheel (Figure 1.4.-A) and left to dry in a cool, dry place without drafts (Figure 1.4.-B).



Figure 1.4. Photographs of A) shaping mud for pottery B) drying conditions of the pots

It is important to avoid drafts, as they can accelerate drying and cause cracks. When the dried pots are ready for firing, they are typically fired in open areas using juniper wood, depending on the season and wind conditions. The juniper wood releases oil as it heats, which is absorbed by the pots, allowing them to dry without cracking. Once the juniper wood burns down to ashes, the pots are placed in the ashes and covered with more juniper wood. The firing process is complete when the upper layer of juniper wood burns down to ashes again, taking approximately 40-45 minutes (Figure 1.5).



Figure 1.5. Final products after baking the pots in field.

1.2. Literature Review

The southern region of Western Anatolia, despite hosting a wide range of clay minerals, is primarily known for its prominent metallic minerals. The region's most ancient rock formations have undergone high degrees of weathering and erosion, resulting in a vast spectrum of clay minerals with varying purity levels. When conducting a regional literature review, Akin's (1988) doctoral dissertation on current pottery production in the Dont plateau stands out as a specific study focusing on the traditional production methods. Momigliano et al. (2011) and Momigliano and Kibaroglu (2017) have conducted archaeological studies in this region. The study determined that four main reference clay sources (Çaltılar, Seki, Söğüt, Gölhisar) were used in ceramic production, and different preparation techniques were applied with these clay sources. The use of various clay sources and additional materials suggests the absence of a standardized production recipe, leading to the conclusion that there was no centralized ceramic tradition in Çaltılar. In another study, Bozyiğit (2020) examined the soils of the Seydikemer district. The findings indicate the presence of various soil types in the region, with analyses revealing that these soils generally contain high carbonate levels and low organic matter content. Local studies do not provide a comprehensive database for Dont clay, but they offer general information regarding the geological structure of the area.

Other studies have primarily focused on the geology of the Esenköy (Dont) region, Dont plateau, and surrounding areas. Dilber (1992) studied the geology and evaluation of industrial rocks in the Günlükbaşı-Üzümlü (Fethiye) regions. Karadağ et al. (2009), examined mineralogical, petrographic, and geochemical properties of chromite formations in the Yanıklar-Çiftlik-Karagedik (Fethiye - Muğla) region. Ersoy (1989) and Ersoy (1992) conducted geological and tectonic research in the southern region, including Altınyayla (Dirmil) and Dont Plateau. The studies of Ersoy (1989) and Ersoy (1992) offer significant contributions to the comprehensive evaluation of the geological and tectonic structure of the study area.

More generally, a review of other studies in a broader sense reveals a multitude of diverse research focusing on different materials and characteristics for industrial uses, employing methodological approaches like this thesis (Belevi clay in the Denizli region - Semiz and Çelik, 2020; Uşak clay - Agayev and Kucukuysal, 2018). In a study

conducted by Özkan (2014) in the Western Anatolia region, the ceramic properties of Karacasu clay were examined using similar methods. Similarly, Abubakar et al. (2014) investigated the ceramic potential of Dabagi clay in Nigeria, while Mousharraf et al. (2011) explored the raw material potential of Bangladesh clay. Abdelmalek (2017) performed a mineralogical characterization of Algerian clay for ceramic purposes. Studies conducted in regions significantly distant from Western Anatolia, such as Ukraine, Bangladesh, Algeria, and Nigeria, exhibit methodological and objective similarities, contributing to the development of this research.

Fletcher et al. (2017) reports the early ceramics in Anatolia with a special reference to Boncuklu Höyük. Öney and Çizer (2014) visited the last female potters from Anatolia and similarities in the pottery-making processes were highlighted. Although there is a large literature on pottery in Anatolia from past to present, studies that comprehensively analyze pottery raw materials by addressing the subject from a multidisciplinary perspective are very rare and this study was carried out in order to fill this gap in the literature. In order to achieve the goals of this study, there is a need to understand the ongoing pottery culture in Dont plateau and to determine the role of the geological elements of the region in pottery production.

1.3. Aim and Scope

It is known that clay materials from the Dont plateau in the Fethiye region have been used for pottery production by the local population for many years. The fact that the clay material found in and around the Dont plateau is traditionally used by the local settlers people for pottery production suggests that this raw material might be suitable for the ceramic industry. This observation serves as the basis for the research hypothesis. In this context, the study explores the suitability of Dont clay's physical, mineralogical, geochemical, micromorphological, and thermal properties as a ceramic raw material. Therefore, the major aim of this thesis is to characterize the raw materials from the Dont plateau in terms of mineralogical, geochemical, thermal and micromorphological properties. In this context, the main objectives are;

- to learn the traditional pottery making procedures from the local people and the literature

- to have the samples from the same locations as local people use for mud of pottery
- to characterize the mineralogical composition of the raw materials and additive materials
- to determine the geochemical composition of the raw materials and additive materials
- to create mud following the same methods as local people use and do thermal treatment at 400, 800 and 1000°C.
- to examine thermally loaded samples mineralogically
- to interpret the findings to evaluate the suitability of the Dont clay for ceramics.
- to compare the results with the similar materials used for pottery.

To achieve these goals, several methodologies were utilized. X-ray diffraction (XRD) analysis and Fourier Transform Infrared Spectroscopy (FTIR) analysis were conducted to determine the mineralogical compositions of the samples. Polarizing microscope was used to examine mineralogically and petrographically the thin sections of the host rocks. Differential Thermal Analysis-Thermogravimetry (DTA-TG) was also used to better understand the minerals and their thermal responses.

1.4. Study Area

Study area is located in the Fethiye district of Muğla province, in southwestern Türkiye. Geographically, it lies to the south of Altınyayla (Dirmil) district in Burdur and to the northeast of Fethiye. The area, formerly known as the Dont plateau, is located 93 km from Fethiye, 145 km from Antalya, and 120 km from Burdur by road, and it is marked on the map in Figure 6. In addition to the Dont plateau, studies were also conducted at the Esenköy location for the clay material found in this region. The Dont and Esenköy are the names given to the primary settlements within the study area. The area previously located near the coastline was referred to as Lower Dont, while the plateau to the northeast was identified as Upper Dont. The region known as Lower Dont was later renamed Esenköy. After 2015, both areas were consolidated and

officially renamed as Dont. In this study, the coastal area is referred to as Esenköy, and the plateau to the northeast is treated as Dont (Anonymous, 2024). Study areas are shown on the map (Figure 1.6.). The Dont plateau is actively used as agricultural land by the local people. The samples were collected from the locations and at certain depths of the profiles from which local potters have the clay material.



Figure 1.6. Location map of the study area including Dont Plateau, Fethiye District, Esenköy District

1.4.1. Physiography of the study area

The study area covers approximately 140 km². Noteworthy elevations within the study area include Kelebekli Mountain (2159 m), the Boncuk Mountains (950m-2160m), as well as Eren Mountain (2863m) and Haciosman Mountain (2440m) situated to the south of the region. The study area is subject to the Mediterranean climate and the impact of the Taurus Mountains, resulting in hot and dry summers and mild and rainy winters. During the summer months, temperatures tend to fluctuate between 25-30 degrees. By contrast, the winter months receive high levels of precipitation and present mild weather conditions, with temperatures ranging from 10-15 degrees. These are the average values in the region. In areas closer to the sea, such as Esenköy, temperature

increases a little more and humidity increases. The prominent water source of the region is the Eşen River, originating from the Akdağlar Mountain and running through the Seydikemer district center, extending over approximately 120 km (Anonymous, 2017).



2. GEOLOGY

2.1. Regional Geology

The study area is located in the western Anatolian region of Türkiye, on the Aegean plate, and the tectonic processes of the Aegean plate directly affect the geological structure of this region. Therefore, it is necessary to first explain the general geological structure of this region with the tectonic evolution processes of the western Anatolia region.

The Aegean region, which stretches from Western Thrace in the north to the Hellenic Arc in the south, is about 800 km long and was formed by tectonic interactions between the African, Arabian, and Eurasian plates (Yılmaz, 2000). The North Atlantic began to form in the middle Jurassic when continental rifts separated Africa from North America, while Eurasia separated from North America in the middle Cretaceous (Higgins and Higgins, 1996). The faster movement of the South Atlantic caused the African plate to rotate clockwise, resulting in the closure of the Tethys Ocean between the African and Eurasian plates. This tectonic activity continued when the Arabian-African plates collided with the Eurasian plates, especially affecting eastern Anatolia in the middle and late Miocene (McKenzie, 1972; Dewey and Şengör, 1979). (McKenzie, 1972; Dewey and Şengör, 1979). The Arabian plate's collision along the Bitlis-Zagros suture zone compressed the Anatolian plate northward (Figure 2.1.), causing westward movement of the Anatolian block and eastward movement of its northeastern part (McKenzie, 1972). As a result, the Anatolian block is displaced westward by the NAF (North Anatolian Fault) and the EAF (East Anatolian Fault). This westward escape is curved toward the Southwest due to continental thickening in Greece, with the movement continuing toward the Hellenic Arc (McClusky et al., 2000; Dewey and Şengör, 1979; Le Pichon and Angelier, 1979, 1981; McKenzie, 1972, 1978; Şengör and Yılmaz, 1981; Taymaz et al., 2007).

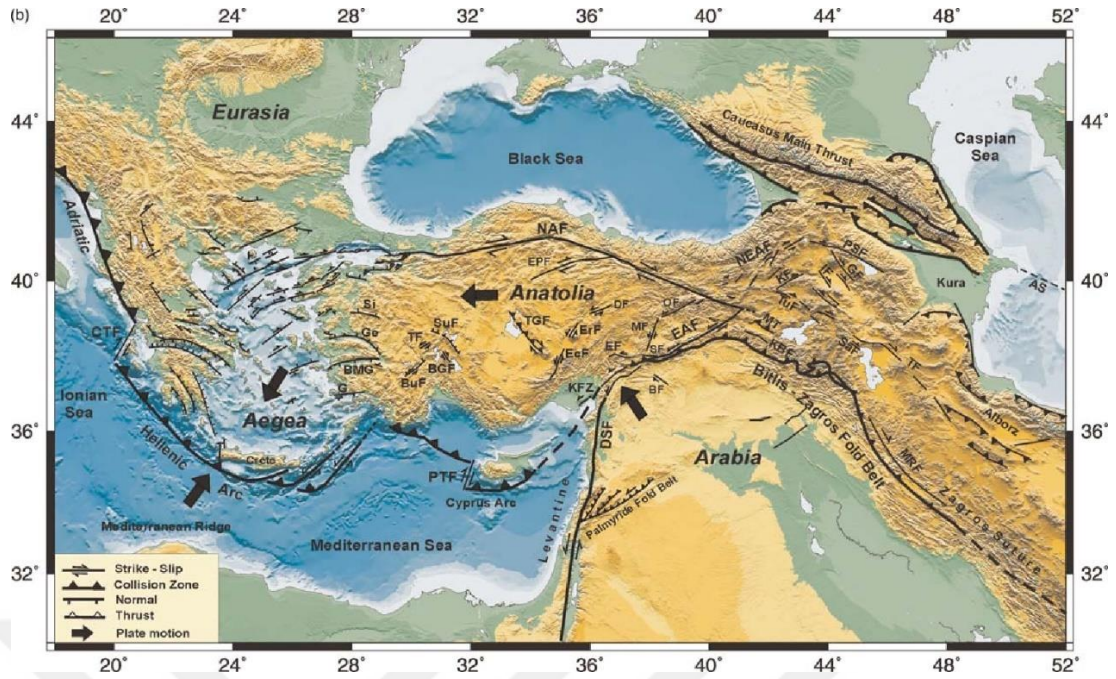


Figure 2.1. Areas of coherent motion and zones of distributed deformation in Aegean region. Heavy arrows indicate generalized regional motions (Taymaz et al., 2007)

This tectonic mechanism led to the gradual extension of western Anatolia. In addition, the subduction of the African plate beneath the Aegean plate along the Greek arc is a key factor in the extensional tectonics of the region. The resulting tectonic activity has formed metamorphic core complexes in Greece and western Anatolia (Charalampakis et al., 2014). Therefore, the active tectonics in the Aegean region has been described as an "extended neotectonic system" (Dewey and Şengör, 1979).

The geological structures of the Mugla region were formed by these tectonic processes. The Taurus Mountains, located in the southern part of the study area, are part of the Alpine-Himalayan belt formed by the closure of the Tethys Ocean and the collision of the African plate with the Eurasian plate. This process began in the late Mesozoic about 100 million years ago and has continued to the present day (Robertson et al., 2012; Şengör and Yılmaz, 1981). The tectonic movements caused by the closure of the Tethys Ocean and the southwestward movement of the Anatolian Plate have formed a complex series of geological structures (Figure 2.2), including various overlying rock formations and thrust systems, strike-slip faults and thrust faults, which were formed at different times and by different mechanisms (Koçyiğit, 1991; Koçyiğit and Özacar, 2003; Ok and Tüysüz, 1999; Robertson et al., 2012; Şengör and Yılmaz, 1981).

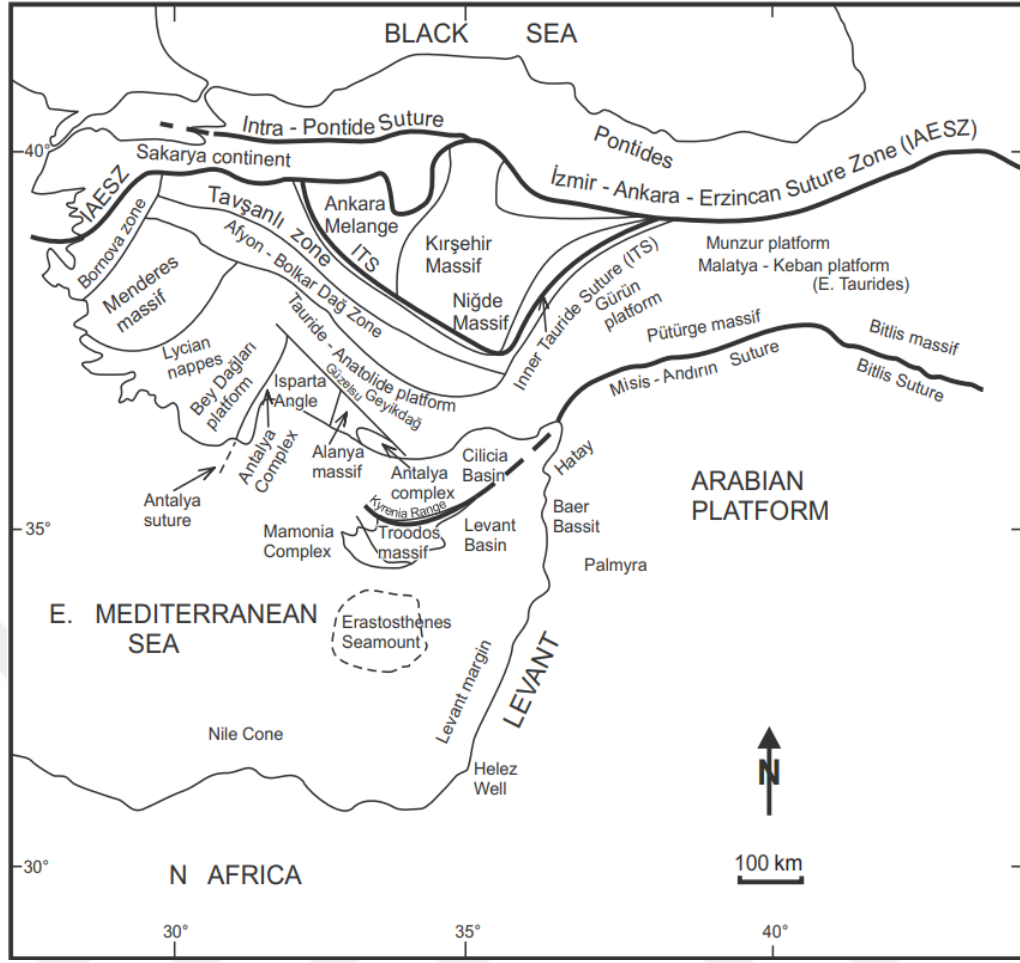


Figure 2.2. Tectonic map of the Anatolian and Eastern Mediterranean region which include main tectonic units (Robertson et al., 2012).

The main tectonic units in the Muğla region, southwest Türkiye, include several key formations. The Anatolide-Tauride Block consists of the Paleozoic clastic-carbonates and Mesozoic carbonates (Okay and Tüysüz, 1999). The Western Taurus has the Paleozoic-Tertiary sedimentary layers formed through Alpine-Himalayan compression and folding (Okay and Tüysüz, 1999). The Menderes Massif, the largest metamorphic mass in Anatolia, emerged during Western Anatolia's expansion (Okay and Tüysüz, 1999; Okay et al., 2001). The Lycian Nappes, lying above the Anatolide-Tauride Block, consist of the Mesozoic carbonates, volcano-sedimentary units, and ophiolites, formed by high pressures from Tethys Ocean closure and African-Eurasian collision (Okay, 1989; Görür et al., 1995; Okay et al., 2001; Gül, 2018). The Beydağları Autochthon, beneath the Lycian Nappes, contains the Upper Cretaceous neritic limestones and Paleozoic metamorphic rocks (Okay, 1989; Okay et al., 2001; Aksoy and Aksarı, 2008; Gül, 2018). The Yeşilbarak Nappes are found between the Beydağları Autochthon and the Taurus Mountains and include the Mesozoic neritic

limestone, sandstone, shale, marble, and the Tertiary flysch-like sediments (Şenel, 2007; Aksoy and Aksarı, 2008; Gül, 2018). The Fethiye-Burdur Fault Zone (FBFZ) is a significant strike-slip fault that played a major role in Western Anatolia's tectonic evolution. These units collectively contribute to the region's complex geological structure.

2.2. Geological Setting of The Study Area

The Dont plateau is located in a broad area that includes young neotectonic sediments, the Yeşilbarak and Lycian nappes to the north of the Beydağları Autochthon (Şenel, 2004). The units containing the clayey weathering products consist of sediments and carbonate rocks of the Triassic to the Paleocene, as well as Lycian Nappes and the Quaternary sediments, which include late Cretaceous-aged ophiolites (Ersoy, 1990; Collins and Robertson, 1997, 1998, 1999). In previous studies, many researchers have examined the region in different sections. Poisson (1977), Poisson (1984) and Alçiçek et al. (2019) examined the Antalya-Burdur-Korkuteli-Isparta in detail and divided the region into three main sections as Beydağı Autochthon, Lycian Nappes and Antalya Nappes (Figure 2.3.).

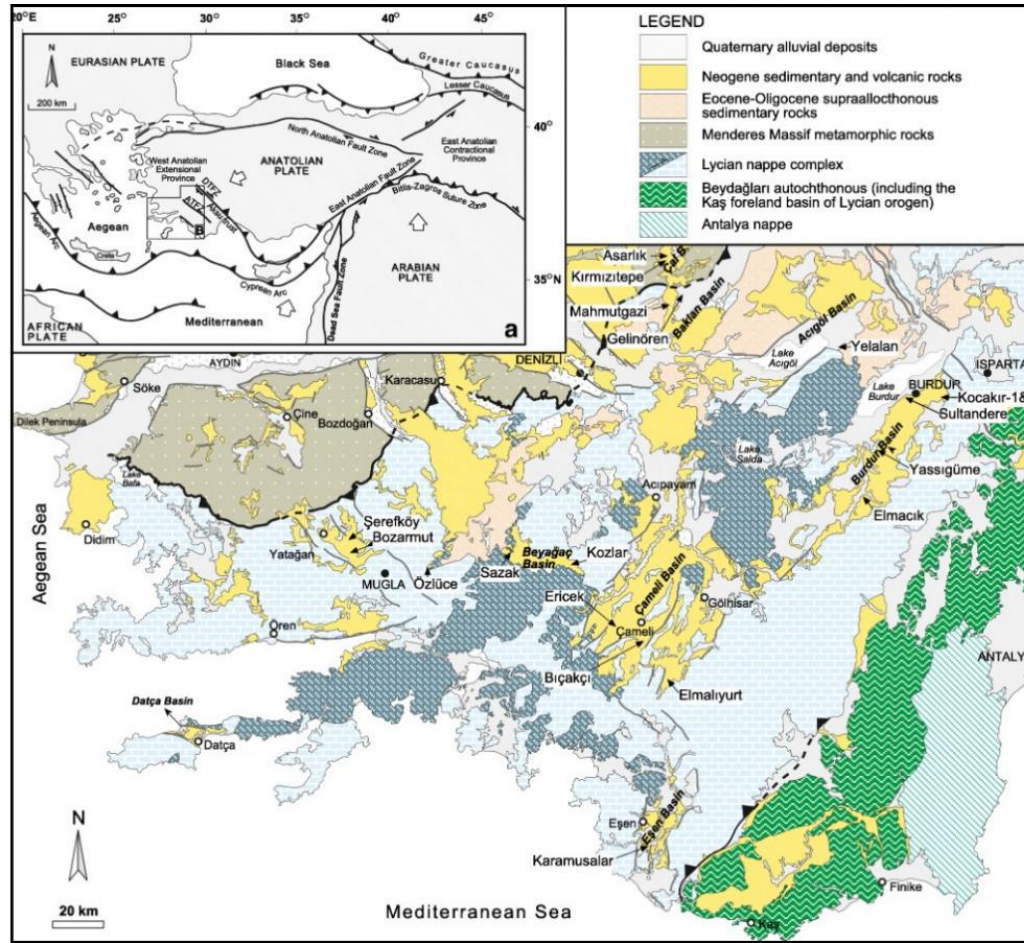


Figure 2.3. Geological map of southwestern Anatolia, including an inset of the major tectonic units of Türkiye (Alçiçek et al., 2019)

Ersoy (1989) includes the Güneydağı group, the Kelebekli Dağ group and the Dirmil Olistostromal Melange units in this region (Figure 2.4.).

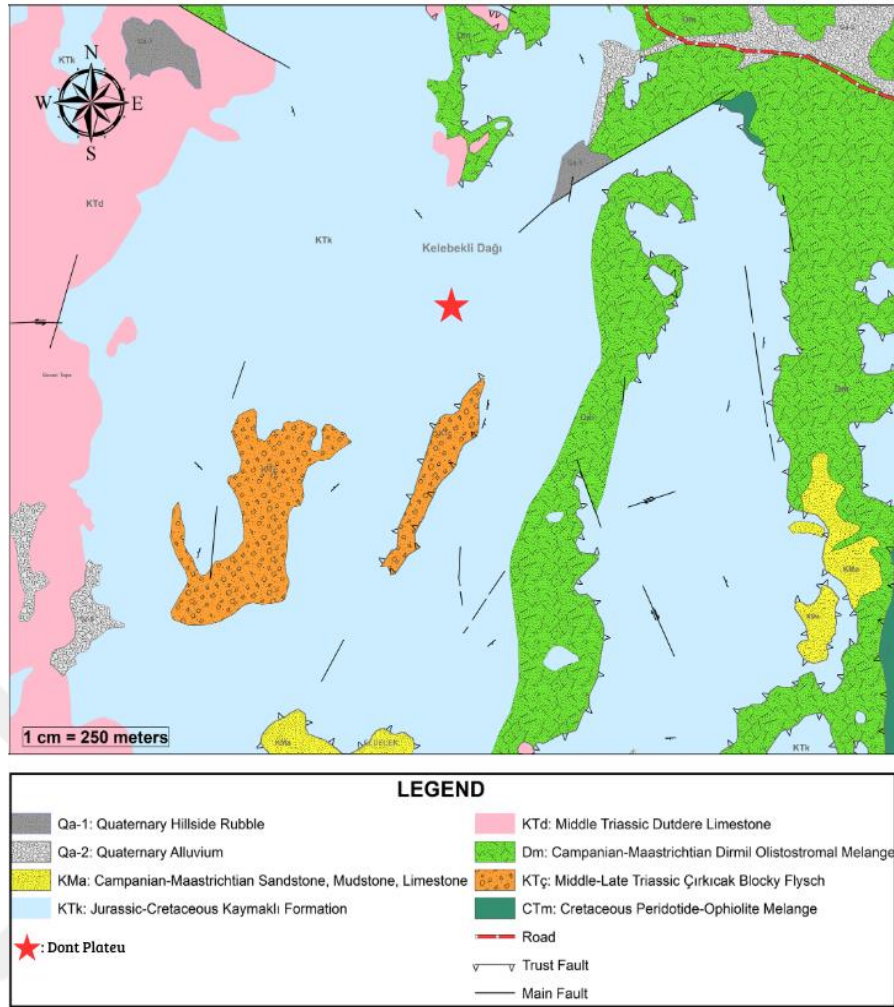


Figure 2.4. Geological map of the study area (Ersoy, 1989).

2.2.1. Stratigraphy

2.2.1.1. Main units

The stratigraphy of the main units is summarized from Ersoy (1989) and showed in Figure 2.5.

Güneydağı Group; The rocks belonging to the Güneydağı group are named after the Güney Mountain in the south of the study area. Çırkıcak Blocky Flysch is included in this group. The unit is the uppermost formation of the Güneydağı group and consists of clastic rocks.

Dirmil Olistostromal Melange; The Dirmil Olistostromal Melange is a sedimentary melange containing fragments and blocks from ophiolites, volcanics and clastic rocks in the region.

Kelebeklidağ Group; The rocks belonging to the Kelebekli Dağ group are named after the Kelebekli mountain region in the study area and consist of Kaymaklı formation and Dutdere limestones. The Kaymaklı formation represents the deep-sea sediments found on the Dutdere limestone. It contains radiolarite cherts in greenish, gray and red colors, spilitic basalts, clay limestones with an ammonites and marl units. In addition, representative samples that taken from the Dont plateau are included in this formation. Dutdere limestones contain dolomitic limestones with a massive appearance of grayish-white color and megalodon fossilled, silty, sandy and pebbled limestones (Ersoy, 1989).

When the Dont plateau is examined stratigraphically, there is the Triassic-Lower Jurassic aged Kelebekli Dağ group at the base and above it is the Upper Cretaceous aged Çırkıcak Blocky Flysch and the Paleocene aged Dirmil Olistostromal Melange, which belong to the Güneydağı group with tectonic contact, respectively. Current sediments in the region are surfaced incompatible with Dirmil Olistostromal Melange (Ersoy, 1989).

UPPER PERIOD	PERIOD	EPOCH	GROUP	FORMATION	SYMBOL	LITOLOGY	DESCRIPTIONS
	QUATERNARY		CURRENT	ALLUVIUM	Qal-Qa		Current Formations
MESOZOIC	PALEOGENE		Dirmil Olistostromal Melange		Ktd		Discordance Sedimentary melange consisting of fragments and blocks of various origin
					Ktd		Tectonic Contact
	KRETASE	LATE	GÜNEYDAĞI	ÇIRKICAK BLOCKY FLYSCH	Ktc		Flysch and elastics
							Tectonic Contact
	JURASSIC	EARLY	KELEBEKLİ DAĞ	KAYMAKLI	Jkk		Deep marine sediments
	TRIASSIC	LATE		DUTDERE	Tkd		Dolomitic limestone

Figure 2.5. Stratigraphic section of Dont plateau and its surroundings (Ersoy,1992)

In the study area, grayish-white dolomitic limestones are observed dominantly along with the brown-coloured Quaternary sediments and sandy-gravelly limestone intercalations. These units, represented by Dutdere limestone and recent formations, contain some clay materials. The brown-coloured sandy-gravelly soil and the white-coloured unit nourished by the parent rock, represent distinct clay layers observed in the region. The evolution of these units, along with the parent rock, continues in the present day through various erosion and transportation processes.



3. MATERIAL AND METHODS

Following the literature review, field studies were conducted. These studies were carried out for the purpose of validating and gaining a deeper understanding of the information derived from the literature, together with the collection of field samples for related analyses. Following the field studies, laboratory studies were initiated, where necessary tests and analyses were performed. Both of these stages of work were conducted to respond to the general objectives and specific research questions of the study and are essential for achieving results aligned with the goals of this study.

In the final stage, the evaluation of the gathered data was carried out within the scope of office studies. At this stage, findings from the field and laboratory studies were discussed in detail and analysed along with the topic. The analyses were interpreted and compared with the results of similar studies conducted in the nearby area.

3.1. Methodology

3.1.1. Field study and description of the samples

The study area is located in Fethiye - Dont plateau, within the O23-a1 and O23-a2 plates of 1/50.000 scaled geological map of Türkiye (Ersoy, 1986). A field study was conducted to gather information from the local community about traditional pottery making and to learn where they obtain the raw materials and how they process them (see Chapter 1, part 1.1.). Following the initial site visit, it was learned that the local community uses multiple materials in pottery making. They use a red-brown clay-sandy-gravelly material as well as a white clay-silty one. The locations where both materials are obtained in the Dont plateau and Esenköy Village were visited, and samples were collected. Following fieldworks were conducted to better understand the geology of the region, including the rock-soil transitions and weathering surfaces. To

understand the parent rock features and relate them to weathering products, samples were also collected from the host rocks outcropping in the region for mineralogical and petrographic analyses. In total, three host rock samples, two clayey samples (Dont clays), one clayey material termed 'additive' from this point onward, and one clayey decoration material were collected (Table 3.1.) and all samples were marked on the relevant map (Figure 3.1.). Dont clay samples collected for both side of the study area (upper Dont and lower Dont) and therefore, one of them referred as “Dont clay” which is sampled from upper Dont and the other one referred as “Esenköy sample” which is sampled from lower Dont (Esenköy village). Both clay materials are known as "Dont clay" and named differently in this study because of their locations.

Table 3.1. List of Samples

Sample ID	Sample	Easting	Northing	Elevation	Description
DC2022L02-M01	Soil	732947	4082945	1224	Dont Clay
DC2022L09-ES	Soil	699498	4055120	138	Esenköy Sample
DC2022L02-	Soil	732947	4082945	1224	Main Additive
DC2022L1-O1	Soil	733358	4082981	1221	Decorational
DC2022L02-SD	Host	732947	4082945	1224	Gabbro
DC2022L08-PBR	Host	695281	4066087	395	Peridotite
DC2022L03-	Host	719725	4072007	1043	Limestone

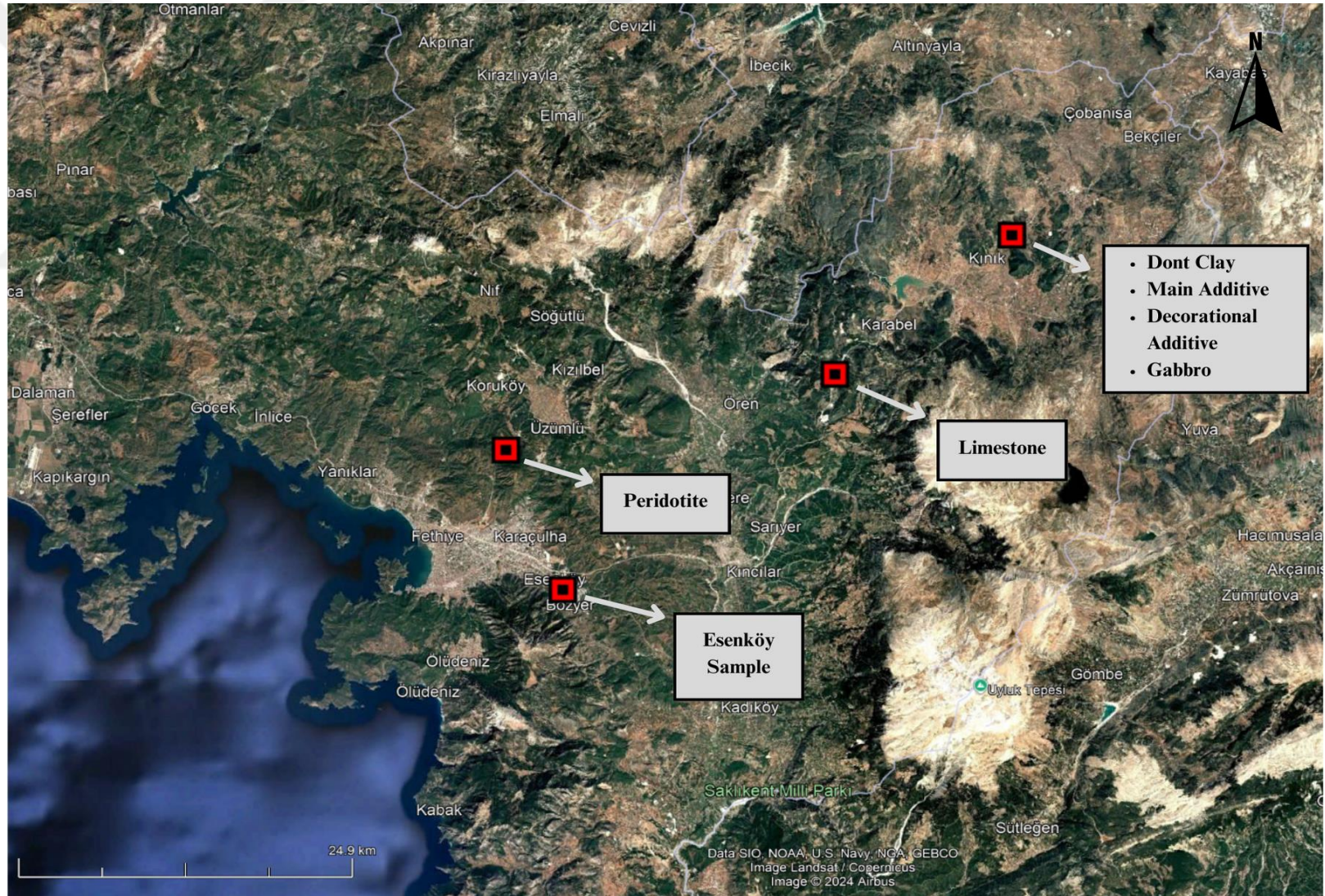


Figure 3.1. Sampling Points in The Field of Upper and Lower Dont plateau

After sampling, the samples were prepared for subsequent laboratory analyses (Figure 3.2.).

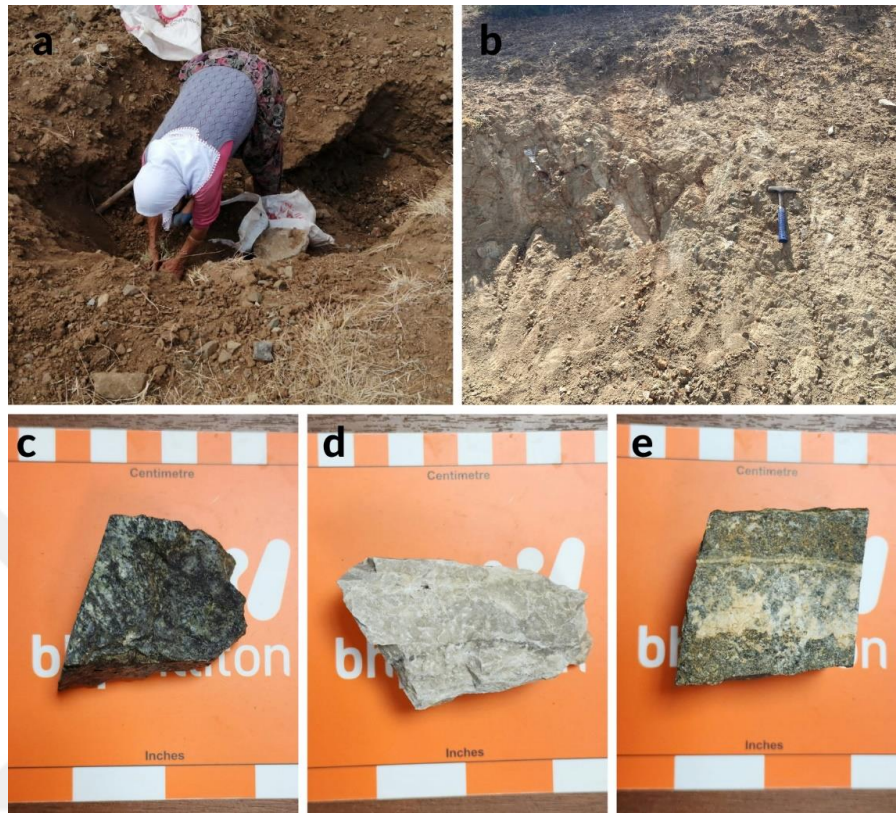


Figure 3.2. Sampled raw materials and parent rocks images. a: Brown coloured soil that contains Dont clay (DC2022L02-M01); b: White coloured additive clay material (DC2022L02-AM01), c: Peridotite sample (DC2022L08-PBR), d: Limestone sample (DC2022L03-DLBR), e: Gabbro sample (DC2022L02-SD).

3.1.2. Analytical studies

The field samples collected for this study were carefully prepared according to the requirements of each analysis. These analyses were divided into three main categories. The first category includes mineralogical analysis, including X-ray diffraction (XRD) analysis. XRD analysis provides a detailed understanding of the crystal structure of the minerals in the sample. Another mineralogical analysis is Fourier transform infrared spectroscopy (FTIR), which also reveals the mineral composition by determining the molecular fingerprint of each sample. Petrographic analysis also happens to study the mineralogical and textural features of the samples and to understand their susceptibility for the chemical weathering. The second type of the analysis was to decipher the chemical composition of the samples. X-Ray

Fluorescence Spectrometry (XRF) was employed to determine the major oxide compositions of the samples. To better understand the technological properties of the samples, Differential Thermal Analysis (DTA)-Thermogravimetric Analysis (TGA) were conducted. All these analyses were carried out in the laboratories of the General Directorate of Mineral Research and Exploration (MTA).

Different types of analyses were conducted on the field samples, considering the objectives and goals of the study. The analyses applied were selected based on the sample type and the data required for characterization. While only XRD and thin-section analyses were performed on rock samples, additional chemical and thermal analyses were carried out on soil samples (Table 3.2.).

Table 3.2. List of analysis carried out in this study.

Host Rocks	Raw Materials	Additives	Ceramic Muds
Gabbro (DC2022L02-SD)	Dont clay (DC2022L02-M01)	Main additive (DC2022L02-AM01)	MM1 (DC2022L1-MM1)
Limestone (DC2022L03-DLRB)	Esenköy sample (DC2022L09-ES)	Decorative additive (DC2022L01-O1)	MM2 (DC2022L02-MM2)
Peridotite (DC2022L08-PBR)			
Mineralogical analysis	Mineralogical analysis	Mineralogical analysis	Mineralogical analysis
• XRD Bulk	• XRD Bulk	• XRD Bulk	• XRD Bulk
	• Clay Fraction	• Clay Fraction	• Clay Fraction
	• FTIR	• FTIR	• FTIR
Physical analysis	Geochemical analysis	Geochemical analysis	Geochemical analysis
• Thin section	• XRF	• XRF	• XRF
	Thermal analysis	Thermal analysis	Thermal analysis
	• DTA-TG	• DTA-TG	• DTA-TG

3.1.2.1. Mineralogical analysis

To determine the mineralogical compositions, the samples were analyzed by a Panalytical Expert Pro diffractometer equipped with a Cu tube at 40kV voltage and 30mA current with a scanning rate of 2°/min. The raw material's X-ray diffraction pattern was recorded between 4° and 70° two-theta angles. For samples separated by sedimentation and centrifugation to obtain the clay fraction, scanning was performed on air-dried, ethylene glycol-saturated, and heat-treated samples at 350°C and 550°C to differentiate the minerals in the clay fraction. Dont clay samples, additives and decoratives as well as the mixtures that are ready for the pottery making (referred to as ceramic mud from this point forward) were all scanned through XRD. Analyses of the bulk fraction, clay fraction, and fired samples were conducted for the Dont clay samples (DC2022L02-M01 and DC2022L09-ES) and the main additive material (DC2022L02-AM01). For the decorative additive (DC2022L1-O1), only XRD bulk fraction analysis was performed. Additionally, both bulk fraction and fired sample analyses were separately conducted for the ceramic muds (DC2022L1-MM1 and DC2022L02-MM2). Minerals were identified according to the methods of Chen (1977) and Moore and Reynolds (1989). Main raw material, additive and decoration material as well as the ceramic muds are all analyzed semi-quantitatively according to the method of Brown and Brindley (1980) based on the intensity factors of the reflections given in the study of Cook et al. (1975).

Fourier Transform Infrared Spectroscopy (FTIR) analysis was carried out by Thermo Scientific iS10 with a resolution of 16 cm⁻¹ and a number of scans 32 cm⁻¹ between 400 and 4000 cm⁻¹ intervals. As happened in XRD analysis, all the samples were examined by using a FTIR spectrometer except decoration additive material.

Thin sections were prepared for petrographic analysis and analyzed in the laboratory of the Geological Engineering Department at Muğla Sıtkı Koçman University using a Leica DM2500 polarizing optical microscope. Mineralogical and petrographic determination of the samples Gabbro (DC2022L02-SD), Limestone (DC2022L03-DLBR) and Peridotite (DC2022L08-PBR) were carried out to better understand the host rock susceptibility for weathering conditions.

3.1.2.2. Chemical analysis

Major oxide compositions of the Dont Clay samples as well as the ceramic mud samples were characterised by using XRF. Loss on ignition values were also determined for the aforementioned samples. The values are listed in weight percentages (wt %).

3.1.2.3. Thermal analysis

The Dont clay samples and main additive sample were all characterised by Rigaku Thermoflex TG 8110 brand DTA-TG at a temperature range from ambient to 1000°C with a heating rate of 10°C min⁻¹ under nitrogen atmosphere.

4. RESULTS

This chapter presents the results of all analyses conducted in this study. It is organized as follows: first, the results of the host rock analyses are detailed. Next, the findings from the analysis of Dont Clay samples are presented, followed by the characterization of materials used as additives. The chapter concludes with the characterization of the ceramic muds.

4.1. Analysis of Host Rocks

Examination of thin sections characterizes the minerals and the basic textures of the host rocks which helps to understand the primary minerals behaviours under weathering conditions. The powders of the two (Gabbro and Limestone) of the three host rocks were analyzed by using XRD. All host rock samples (Gabbro, Limestone and Peridotite) were analyzed under polarizing microscopy. Mineral abbreviations are given according to Whitney and Evans (2010).

4.1.1. Petrography of host rocks

Three representative host rocks were sampled from the study area. They are the possible sources of the Dont clay. The first host rock is a gabbro sample, and the hand specimen of this sample has dark green to grey color and reveals phaneritic texture and argillic alteration (Figure 4.1.-A). Another host rock is a limestone that sampled from 15 km SW of Dont clay location. Hand specimen of limestone has a light grey color and dolomitic - sugary texture (Figure 4.1.-B). Another host rock is a peridotite that sampled from approximately 8 km NE of Fethiye district. Hand specimens of peridotite have dark grey to black color and phaneritic texture. Serpentinization is also visible in hand specimen (Figure 4.1.-C).



Figure 4.1. Hand specimens of Host Rocks. A: Gabbro (DC2022L02-SD); B: Limestone (DC2022L03-DLBR); C: Peridotite (DC2022L08-PBR)

Thin sections were prepared from all three host rocks and analysed under polarized microscope. The thin section analysis of gabbro sample (Figure 4.2.) reveals that it is predominantly composed of clinopyroxene and orthopyroxene. Notably, chlorite alteration is observed along the edges of the pyroxene crystals. The pyroxene crystals are predominantly subhedral to anhedral in shape, exhibiting low birefringence color from light green to dark green. These crystals display moderate relief and oblique extinction. Chlorite alterations have spread from the edges of other minerals to the interiors and have been a factor that makes mineral identification difficult. Also, there are some opaque and plagioclase minerals in the composition. The surfaces of the plagioclase crystals exhibit a dirty appearance indicative of kaolinization. The composition of the limestone sample (Figure 4.2.) consists of dominantly dolomite and calcite. The peridotite sample's composition have large amounts of olivine and orthopyroxene minerals with high relief, parallel extinction, and second-third order birefringence. In addition, serpentinization surrounding these minerals in the form of veinlets are observed (Figure 4.2.). Serpentinized areas, which are quite numerous, are seen on almost the entire rock surface. In addition, opaque minerals varying in size have also been seen in this rock.

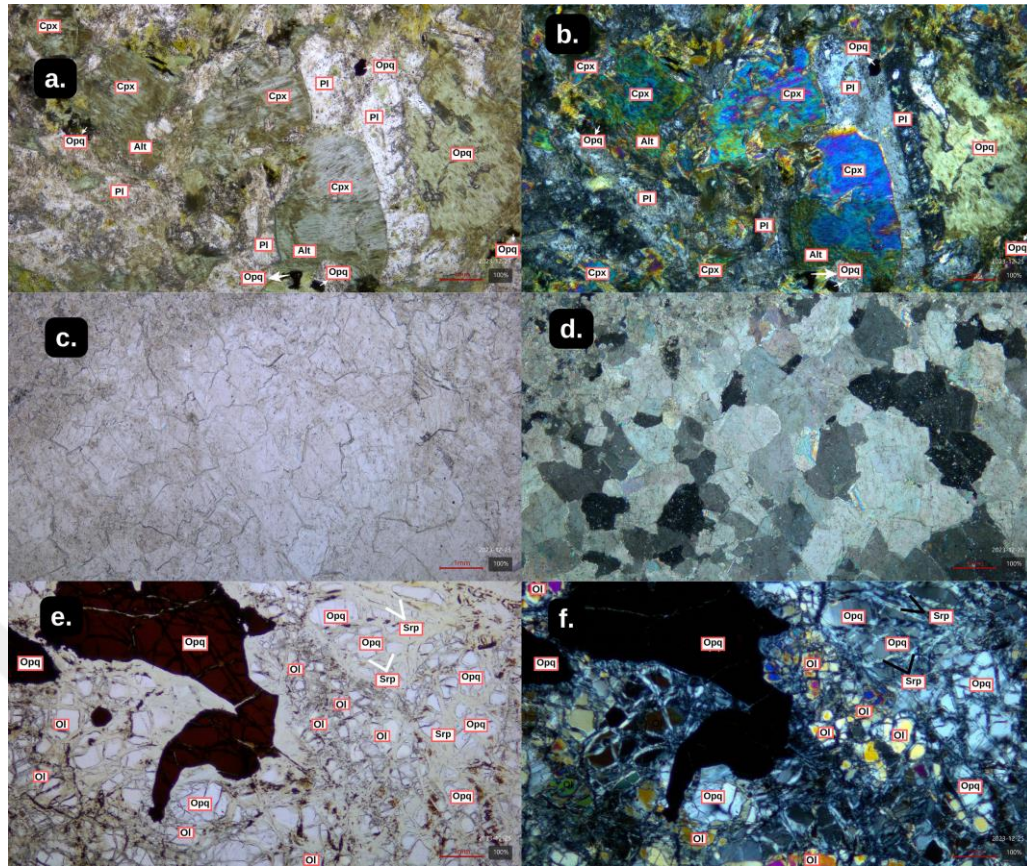


Figure 4.2. Thin section photomicrographs of basement rock samples from Dont plateau and around. (a) PPL and (b) XPL images are sections of the gabbro sample; (c) PPL and (d) XPL images are sections of the limestone sample; (e) PPL and (f) XPL images are sections

4.1.2. Mineralogy of host rocks

Gabbro Sample

In the immediate vicinity of the study area where the Dont clay was found, some host rocks that could be the source of this material were sampled. One of these samples are gabbro and this is sampled from in-situ rubbles around the location of Dont clay. The sample taken was subjected to the necessary procedures for XRD analysis. The bulk X-ray diffraction analysis of the gabbro sample is shown in Figure 4.3. Talc, tremolite, lizardite, calcite, plagioclase and dolomite are identified minerals for describing the composition of the sample. The reflections of $8.83\text{\AA}$ at $9.45^\circ 2\theta$, $4.56\text{\AA}$ at $19.33^\circ 2\theta$, $3.11\text{\AA}$ at $28.60^\circ 2\theta$ and $2.44\text{\AA}$ at $37^\circ 2\theta$ reveal the presence of talc in the bulk composition. Tremolite appears with peaks of $8.34\text{\AA}$ at $10.50^\circ 2\theta$, $3.27\text{\AA}$ at $27.30^\circ 2\theta$ and $2.70\text{\AA}$ at $33.17^\circ 2\theta$. Lizardite shows reflections of $7.93\text{\AA}$ at $11.20^\circ 2\theta$, $3.47\text{\AA}$ at $25.50^\circ 2\theta$ and $2.52\text{\AA}$ at $35.50^\circ 2\theta$ and $2.16\text{\AA}$ at $42.10^\circ 2\theta$. Major reflections of

dolomite at 2.88Å (30.90° 2θ), 2.67Å (33.50° 2θ) and 2.16Å (42.10° 2θ) are also observed in the diffractogram of the bulk composition. Additionally, calcite is determined with the major peaks of 3.04Å at 29.37° 2θ together with 3.86Å at 23.0° 2θ and 2.28Å at 39.50° 2θ and 2.10 Å at 43.0° 2θ. The reflection of 3.19Å at 28.10° 2θ coincides with the plagioclase's major reflection. The reflections of 3.17, 2.87, 2.52 and 2.45 refer to Mg-bearing pyroxene (enstatite?).

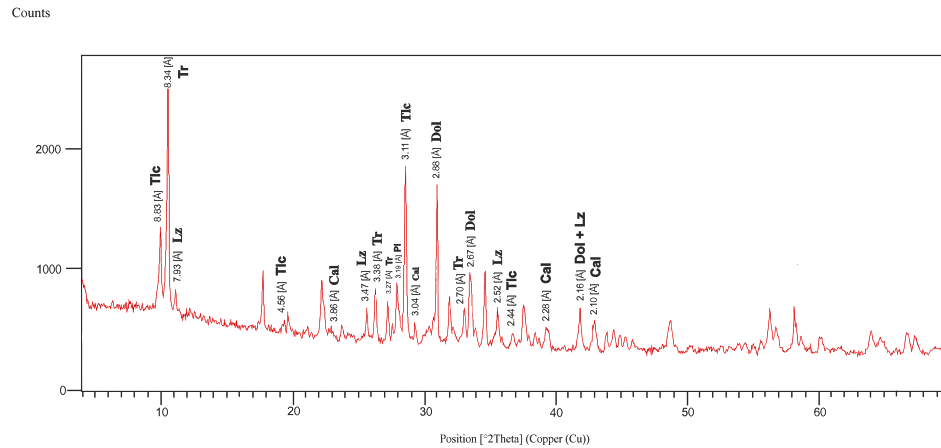


Figure 4.3. XRD bulk results of gabbro sample - DC2022L02-SD (Tlc: Talc, Tr: Tremolite, Lz: Lizardite, Cal: Calcite, Pl: Plagioclase, Dol: Dolomite).

Limestone Sample

The other host rock from the study area is a limestone and it has taken from the outcrop, which is approximately 15 km SW of the location where the Dont Clay was found. The diffractogram of the bulk XRD analysis of the limestone sample is shown in Figure 4.4. The most intense reflection appears at 31.0° 2θ with 2.88Å as dolomite present in the bulk composition of limestone. Relative to the intense dolomite reflection, calcite is observed with a weak reflection of 3.03Å at 29.40° 2θ. Reflections of 8.44Å (10.50 2θ) and 3.12Å (28.80 2θ) refer to the amphibole.

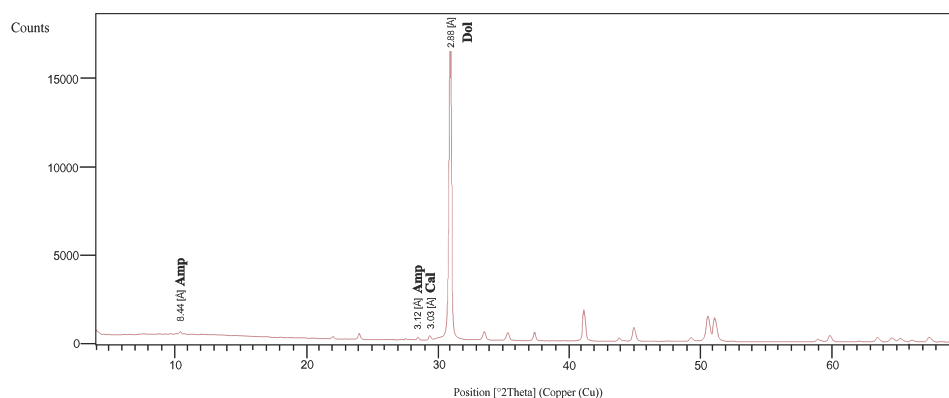


Figure 4.4. XRD bulk results of limestone sample - DC2022L03-DLBR (Amp: Amphibole, Cal: Calcite, Dolomite).

4.2. Characterization of Raw Materials

Raw materials of this study are the Dont Clays from two different locations one from Dont plateau and the other from Esenköy. These two samples have been the main materials for the traditional and modern pottery production. To have a comprehensive characterization of these clay materials, XRD and FTIR analysis were performed to understand the mineralogy; XRF was utilized to reveal the major oxides in the bulk compositions and DTA-TG was used to determine the thermal durability and the phase changes upon thermal loading. Results of these analyses are given below in the same order.

4.2.1. Mineralogy of raw materials

XRD analyses were conducted on both bulk and clay fractions of the Dont Clay samples. Clay fractions of the samples were X-rayed in air-dried, ethylene-glycol solvated and heated to 350° C and 550° C conditions. Additionally, bulk powders of each sample were thermally loaded at 400° C, 800° C and 1000° C with 200° increments every 30 minutes.

4.2.1.1. Mineralogy of Dont clay sample (DC2022L02-M01)

The bulk XRD pattern of the DC2022L02-M01 sample, representing Dont clay, is shown in Figure 4.5. At the low angle's side of the diffractogram, 14.38 Å reflection appears. The reflections attributed to talc are identified at 9.4Å, 4.52Å, 3.12Å (associated with tremolite), 2.59Å, and 2.51Å (associated with kaolinite). Sharp and intense peaks of tremolite appear at 8.4Å, 3.28Å, 3.12Å, and 2.71Å. Reflections of 3.23Å, 2.94Å, and 2.56Å are associated with the presence of pyroxene. Major reflection of plagioclase at 3.18Å together with 4.03Å, and 3.66Å. Quartz major reflection is observed at 3.34Å. Dolomite exhibits 2.98Å, 2.16Å, 2.01Å, and 1.75Å reflections while calcite appears at 3.03Å. Kaolinite mineral displays reflections at 7.28Å, 3.60Å, and 2.51Å.

Semi-quantitative analysis of the Dont Clay sample reveals that amphibole comprises 34% of the total composition, followed by significant amounts of pyroxene (20%) and plagioclase (15%). The presence of kaolinite (7%) and minor amounts of calcite and dolomite is also detected.

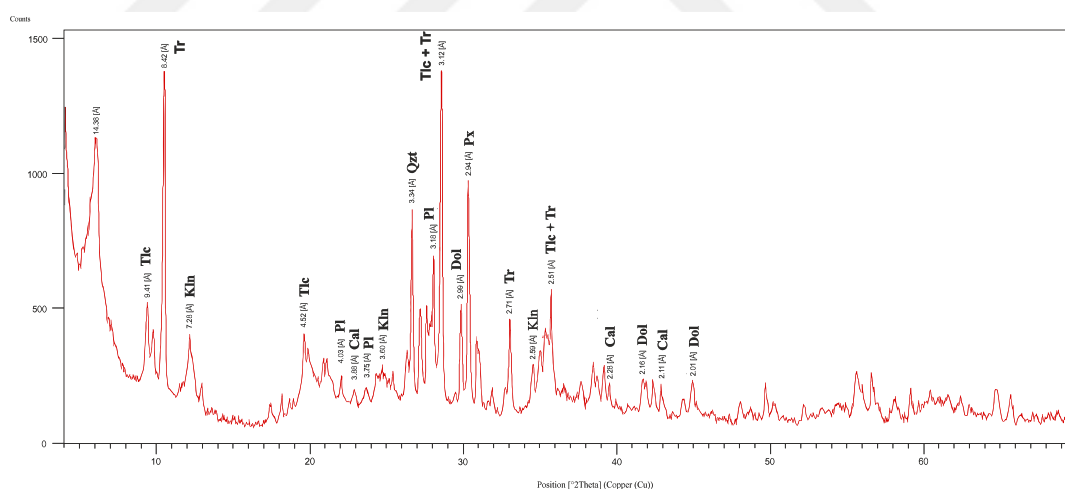


Figure 4.5. Bulk XRD diffractogram of Dont clay sample - DC2022L02-M01 (Tlc: Talc; Kln: Kaolinite; Pl: Plagioclase; Cal: Calcite; Px: Pyroxenite; Tr: Tremolite; Dol: Dolomite)

The diffractograms of the XRD analysis of the clay fraction of the Dont clay sample is shown in Figure 4.6. 14Å reflection in air-dried partially shifts to 16.5Å-17Å upon ethylene glycol solvation revealing the presence of smectite in the clay fraction. Intensity of the non-swelling component (vermiculite) of this 14Å reflection diminishes and collapses into a weak and broad reflection of 10Å by heating to 350 °C and 550 °C.

A wide reflection at 7.3Å indicates the presence of more than one mineral which can be resolved by examining the EG and heated diffractograms. One portion of this peak disappears upon heating at 550°C, clearly identifying kaolinite. The other shoulder is stable under EG and heating treatments corresponding to vermiculite. Talc and tremolite reflections are stable under each treatment and appear on each diffractogram at their original 2 theta angles.

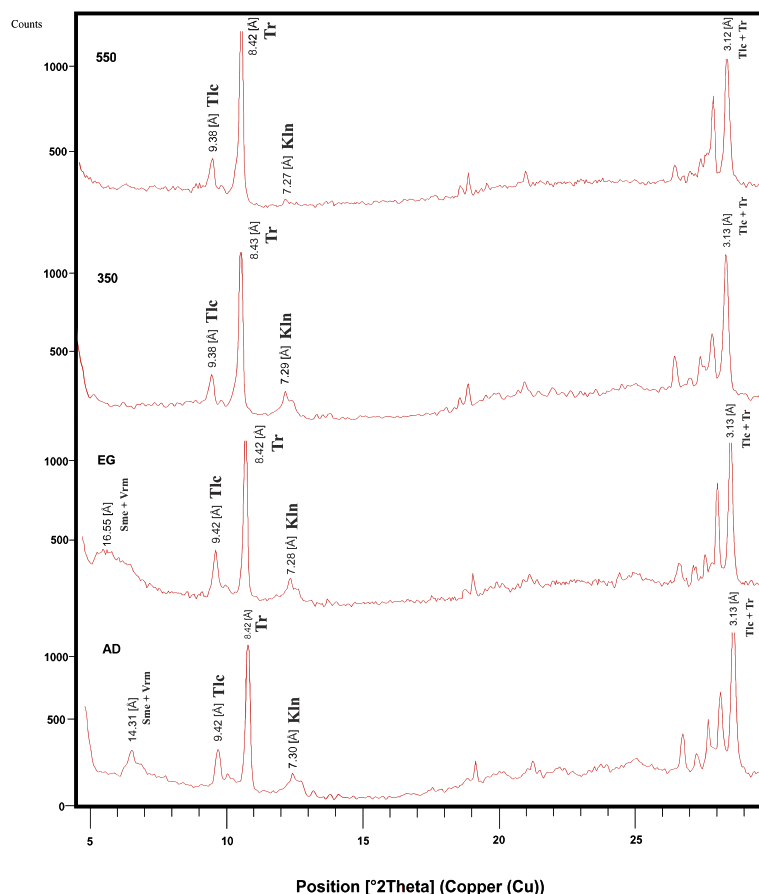


Figure 4.6. XRD diffractogram of clay fraction of Dont clay sample - DC2022L02-M01 (AD: air-dried; EG: ethylene-glycol; 350: heated to 350 °C; 550: heated to 550 °C; Tlc: Talc; Tr: Tremolite; Sme: Smectite; Vrm: Vermiculite; Kln: Kaolinite)

Raw material samples were subjected to XRD analyses by heating up to different temperatures. Each analysis reveals the state of a sample at various phases. In this context, a comparative evaluation was conducted on the XRD bulk diffractograms of the DC2022L02-M01 sample, both bulk normal and heating at different temperatures. The results shown in Figure 4.7. indicate that the intensity of the tremolite peak at 8.42 Å decreased at 400°C and 800°C, but the peak collapsed at 1000°C. Peaks for talc also

collapsed at 1000°C and a new recrystallization phase occurred at 4.09 Å, likely corresponding to cristobalite.

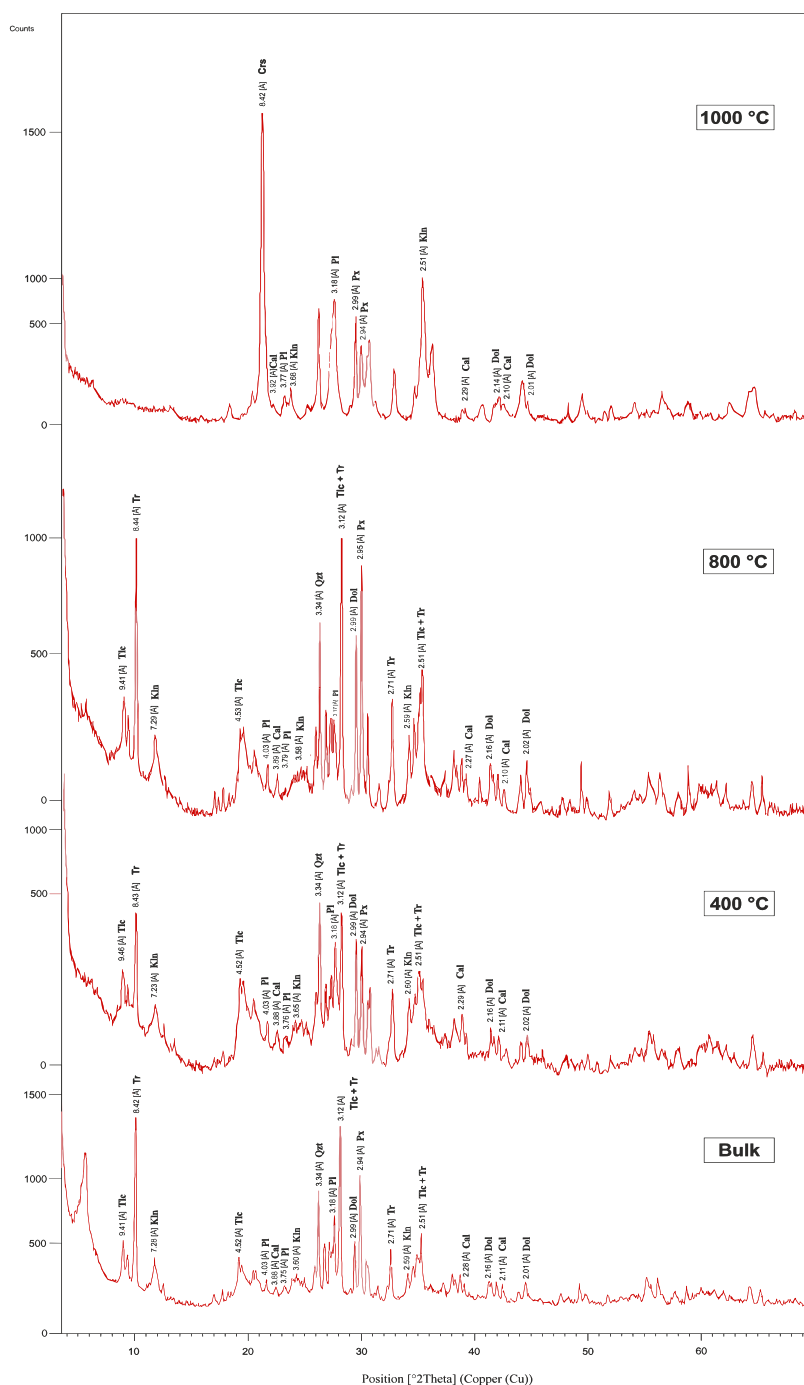


Figure 4.7. XRD Heated Bulk Result of Dont clay sample (Tlc: Talc; Kln: Kaolinite; Pl: Plagioclase; Cal: Calcite; Crs: Cristobalite; Px: Pyroxenite; Tr: Tremolite; Dol: Dolomite)

FTIR analysis of Dont clay sample (Figure 4.8.) shows a broad band at 3373.82 cm^{-1} indicates the stretching vibrations of hydroxyl (O-H) groups, which is attributed to the water content present within the clay structure. The peak at 1637.34 cm^{-1} corresponds

to the deformation vibrations of structural water and hydroxyl groups (O-H) absorbed in the clay minerals. The band at 909.20 cm^{-1} is characteristic of kaolinite mineral (Si-O-Al). The peak at 753.56 cm^{-1} indicates vibrations of the Al-OH-Al compound, while the peak at 686.13 cm^{-1} corresponds to Si-O-Si vibrations (Madejová, 2003; Jozanikohan and Abarghoeei, 2022). The peaks at 507.16 cm^{-1} and 414.83 cm^{-1} are indicative of vibrations from other silicates and metal oxides (Farmer, 2000).

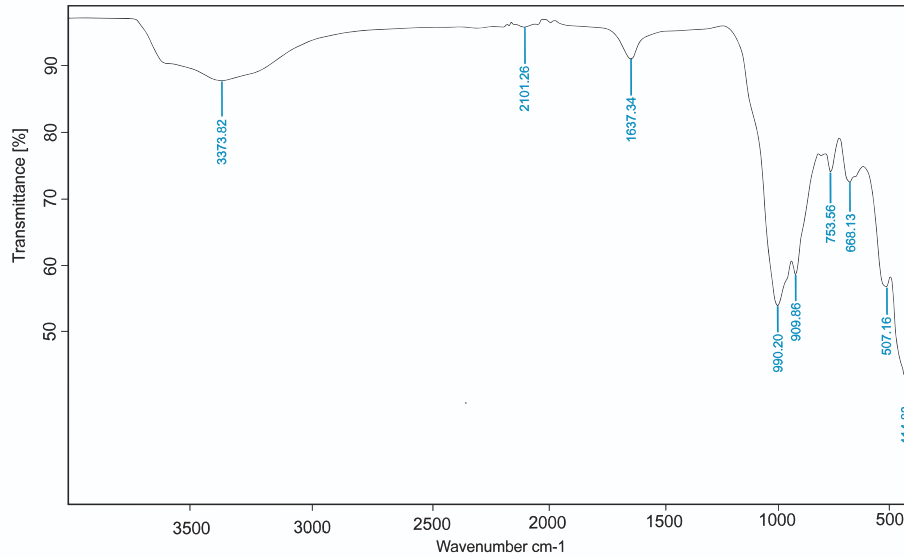


Figure 4.8. FTIR Spectrum of Dont Clay Sample (DC2022L02-M01)

4.2.1.2. Mineralogy of Esenköy sample (DC2022L09-ES)

The bulk diffractogram of the Dont Clay sample from Esenköy presents a composite reflection having a centered peak of 15.2Å at around $5.2^\circ 2\theta$ (Figure 4.9.). Similar to the Dont clay sample from Dont Plateau, talc appears with weak reflections of 4.5Å and 3.12Å ; while amphibole (tremolite) is defined by its prominent peaks of 8.4Å , 3.28Å , 3.12Å and 2.71Å . Additionally, reflections of 2.95Å and 2.57Å are associated with the presence of pyroxene. Plagioclase is observed at 3.18Å . Kaolinite is found with 7.28Å and 3.60Å reflections. Quartz and calcite exist with intense and sharp peaks at 3.34Å and 3.03Å , respectively (Figure 4.9.). The Esenköy Clay sample features a higher proportion of kaolinite (18%) and smectite (16%), reflecting its clay-rich nature, while amphibole and pyroxene are less pronounced (10% and 18%, respectively).

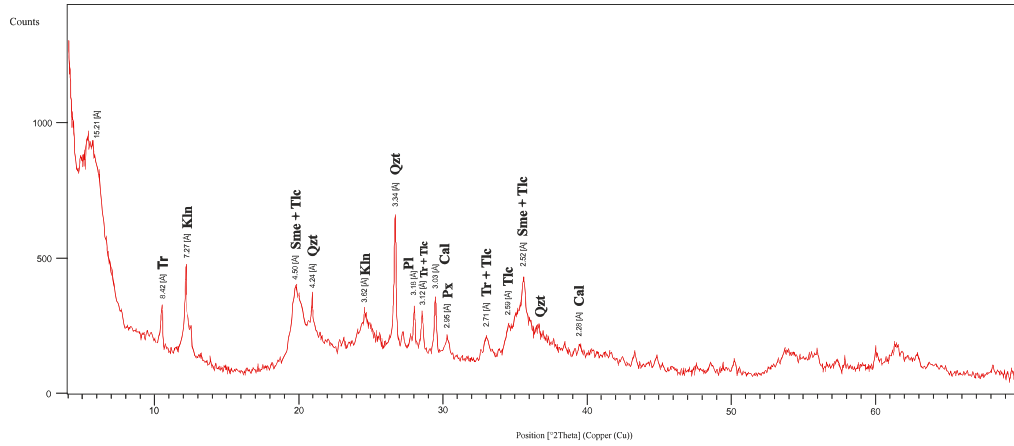


Figure 4.9. The bulk XRD diffractogram of Esenköy sample (Tlc: Talc; Tr: Tremolite; Sme: Smectite; Qtz: Quartz; Kln: Kaolinite; Pl: Plagioclase; Cal: Calcite; Px: Pyroxenite).

The clay fraction XRD analysis of Esenköy sample-(DC2022L09-ES) reveals the minerals in the fine fraction (Figure 4.10.). The 14Å peak in the air-dried XRD diffractogram is a composite and wide reflection pointing to the presence of different 14Å minerals. Upon EG solvation, this peak partially shifts to 16.5Å and collapses to 10Å when heated to 350°C and 550°C reflecting the presence of smectite. On the other hand, the stable part of this 14Å reflection stays stable under EG-solvation and heating treatments and is associated with dehydroxylation of mixed-layered phase.

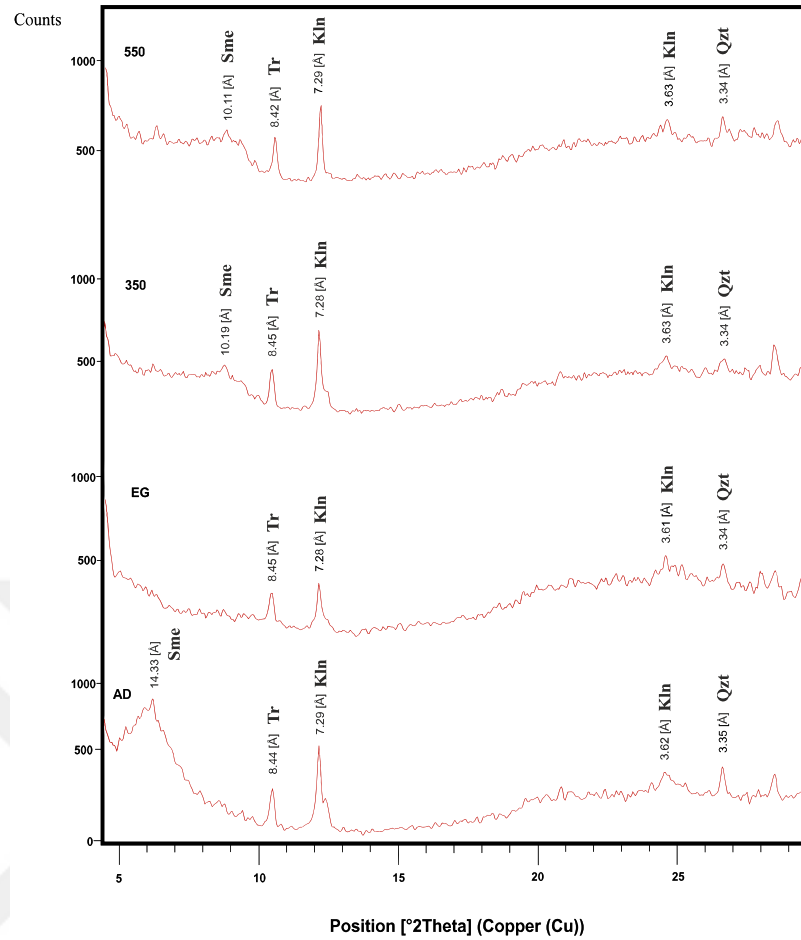


Figure 4.10. XRD diffractogram of clay fraction of Esenköy sample (AD: air-dried; EG: ethylene-glycol; 350: heated to 350 °C; 550: heated to 550 °C; Tlc: Talc; Tr: Tremolite; Sme: Smectite; Qtz: Quartz; Kln: Kaolinite)

The non-clay minerals of the bulk composition of the Esenköy sample (DC2022L09-ES) appear on each diffractogram with decreasing intensities by increasing heating temperature (Figure 4.11.). Basal reflections of the clay minerals disappear upon dehydroxylation at 800°C. Intensity of tremolite reflections totally collapses at 1000°C as does talc so new crystallization occurs at 1000°C. The new phase mineral observed at 4.08Å is likely cristobalite.

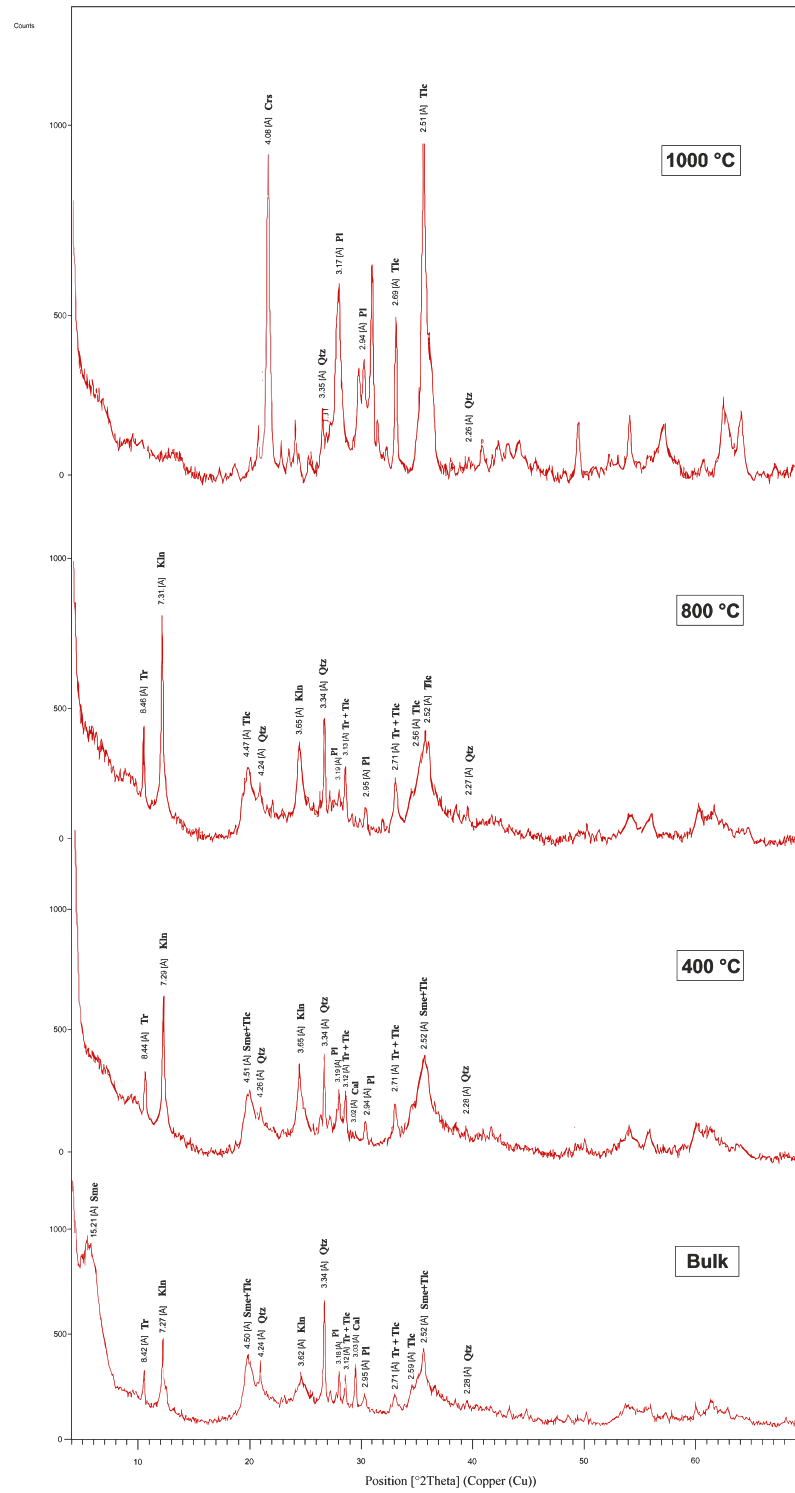


Figure 4.11. XRD Heated Bulk Result of Esenköy Sample (DC2022L09-ES) (Tlc: Talc; Tr: Tremolite; Kln: Kaolinite; Smec: Smectite; Qtz: Quartz; Cal: Calcite; Pl: Plagioclase; Crs: Cristobalite).

Analysis of the FTIR spectrum results of Esenköy sample (DC2022L09-ES) was found to give the same results as observed in the Dont clay sample (Figure 4.12.). Six distinct

peaks were obtained. The first peak, at 3367.33 cm^{-1} , is assigned to the stretching vibrations of hydroxyl (O-H) groups. The band at 1630.26 cm^{-1} is attributed to the bending vibrations of hydroxyl groups and/or structural water incorporated in the clay minerals (Madejová, 2003). The two first peaks therefore indicate that the clay minerals' water content contributes to the vibrational modes observed. The peak at 983.06 cm^{-1} is due to the Si-O-Si stretching vibrations. Finally, the last two peaks are located at 817.72 and 752.04 , respectively, in connection with either aluminium oxide (Al-O), or aluminosilicate (Al-Si-O). The sharp absorption observed at about 752.04 cm^{-1} is assigned to the Al-OH-Al vibrations while that at 606.65 cm^{-1} is attributed to Si-O-Si bending vibrations (Madejová, 2003; Jozanikohan and Abarghoeei, 2022). On the whole, the existence of hydroxyl groups and, at different places, silicate structures represents features typical of clay minerals.

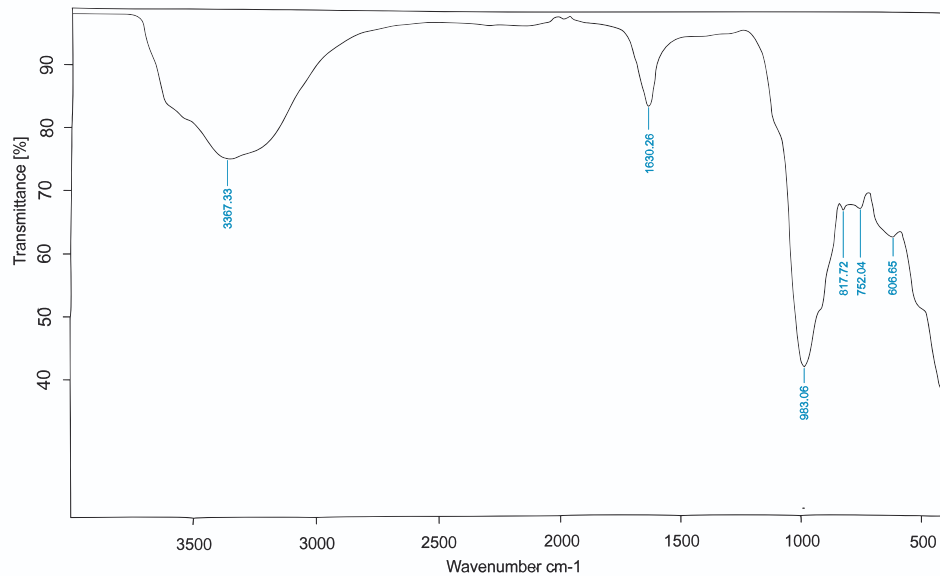


Figure 4.12. FTIR Spectrum of Esenköy Sample (DC2022L09-ES)

4.2.2. Geochemistry of raw materials

Major oxide compositions and loss on ignition (LOI) values of the two Dont clay samples are documented in Table 4.1. High SiO_2 content of both samples (50.8-44.33%) and relatively low content of Al_2O_3 between 11.4-12.1% resemble that the samples have low proportions of kaolinite in the bulk composition (Dondi et al., 2001). The DC2022L02-M01 sample has high content of alkaline earth elements: CaO with 8.5% and MgO with 11.8%. The DC2022L09-ES sample, on the other hand, contains

less CaO (2.7%) but relatively higher MgO (8.2%). Alkaline earth elements are sourced from the host rocks; dolomitic limestone and ultramafics peridotite and gabbro. Total content of alkaline elements is less than 1% for both samples. As expected, Fe₂O₃ content is high (9.6%) for DC2022L02-M01 and higher (19.4%) for DC2022L09-ES. Silicate minerals rich in alkaline earths like smectite, talc, amphibole and chlorite are associated with the major oxides of SiO₂, MgO and CaO. Fe-bearing minerals such as pyroxene, chlorite and hematite are the sources of high Fe content in the bulk composition. LOI values are 6.45% and 10.15% for the samples and closely associated with the mineralogical content of the bulk compositions. Dehydroxylation of hydrated minerals, organic content and decomposition of carbonates increase the LOI for both samples.

Table 4.1. Major oxide compositions of raw materials

Sample ID	Dont clay (DC2022L02-M01)	Esenköy sample (DC2022L02-M01)
LOI (%)	6.45	10.15
Al ₂ O ₃ (%)	11.4	12.1
CaO (%)	8.5	2.7
Fe ₂ O ₃ (%)	9.6	19.4
K ₂ O (%)	0.3	0.3
MgO (%)	11.8	8.2
MnO (%)	0.2	0.3
Na ₂ O (%)	0.4	0.6
P ₂ O ₅ (%)	< 0.1	< 0.1
SiO ₂ (%)	50.8	44.33
TiO ₂ (%)	0.3	0.6

4.2.3. Thermal analysis of raw materials

Thermal durability and the phase stabilities of the Dont Clay samples are examined by Differential Thermal Analysis (DTA)-Thermogravimetry (TG).

Upon examining the DTA-TG graph of the Dont clay sample DC2022L02-M01 is shown in (Figure 4.13.), we observe four distinct endothermic peaks. The first

endothermic peak is located at 111.145 °C, indicating a dehydration process. At this stage, pore water, surface water, or interlayer water is removed from the system. Due to the pronounced depth of this peak, it may indicate the presence of a mineral with high water retention capacity, such as smectite or talc. The second endothermic peak occurs at 501.808 °C, signifying the onset of the first dehydroxylation. This is the stage where hydroxyl groups start to separate from the system, and it continues until the third endothermic peak at 841.671 °C. During this phase, it can be inferred that hydroxyls are still being detached from some minerals. The final endothermic peak occurs at 998.6 °C, marking the completion of dehydroxylation. When the temperature reaches approximately 1000 °C, an exothermic peak is observed, indicating a recrystallization process at this point. Looking at the overall graph, the total mass loss was determined as 7.9 mg.

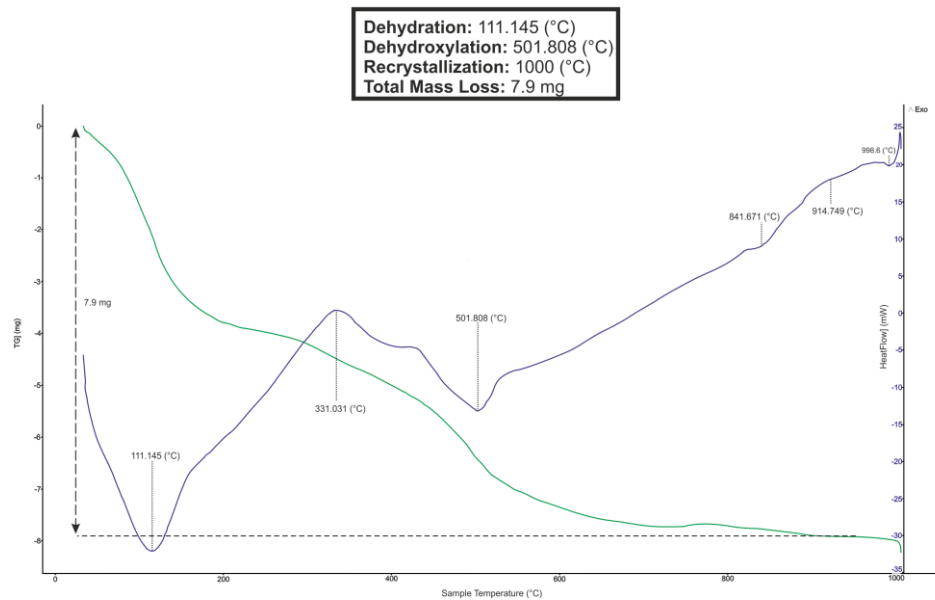


Figure 4.13. DTA-TG graph of Dont Clay Sample (DC2022L02-M01)

The Esenköy sample (DC2022L09-ES) despite being distanced, exhibits results similar to those of the Dont clay sample (DC2022L02-M01). In the DTA-TG thermogram of the DC2022L09-ES sample (Figure 4.14.), there are three endothermic peaks. The first endothermic peak, where dehydration occurs, is observed at 125.869 °C, forming a deeper peak than that of the Dont clay sample. Second and a weak endothermic peak, where dehydroxylation starts, is observed at around 510 °C. Third endothermic reaction happens around 827°C reflecting the last stage of dehydroxylation. The small exothermic peak occurring at approximately 1000 °C

suggests that recrystallization takes place. The total mass loss of the Esenköy sample (DC2022L09-ES) is determined to be 16.8 mg.

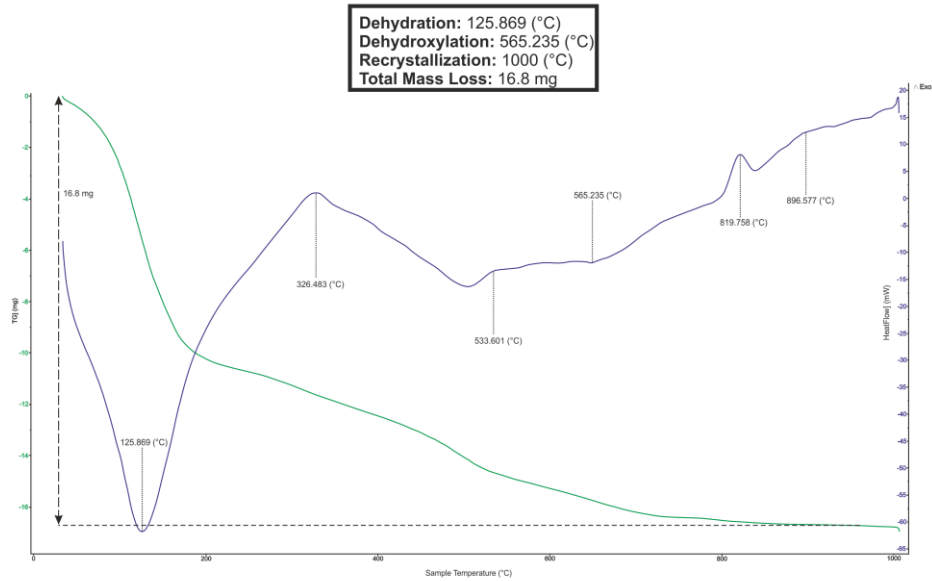


Figure 4.14. DTA-TG graph of Esenköy Sample (DC2022L09-ES)

4.3. Characterization of Additive Materials

There are two different materials both named as additives; one is used as an additive to create the ceramic mud, the other one is used for the decoration. Comprehensive mineralogical, geochemical and thermal characterization of the additives is given in this subchapter.

4.3.1. Mineralogy of additives

4.3.1.1. Mineralogy of main additive material (DC2022L02-AM01)

In the XRD bulk analysis of the main additive material, two reflections with high d-spacing values were initially observed at 21.85Å and 14.53Å, respectively. The 21.85Å reflection refers to the presence of mixed-layered minerals. At the low angle side of the diffractogram, moderately intense and sharp reflection appears at 14Å. Talc also exists in the main additive material with its prominent peaks of 9Å, 4.53Å and 3.11Å. Quite similar to the Dont clay samples, amphibole (tremolite) shows up with 8.4Å,

3.28Å and 2.71Å. Additionally, 7Å reflection is a composite one including more than one mineral resolved by clay fraction analysis. At 2.89Å, dolomite shows a clear reflection. Pyroxene reflections are also identified at 3.23Å, 2.99Å, 2.57Å, and 2.51Å (Figure 4.15.).

Tremolite forms the highest proportion (48%) followed by pyroxene (29%). Talc (7%) and kaolinite (7%) together with plagioclase (5%) and dolomite (4%) form the main fluxing agents of the ceramic body.

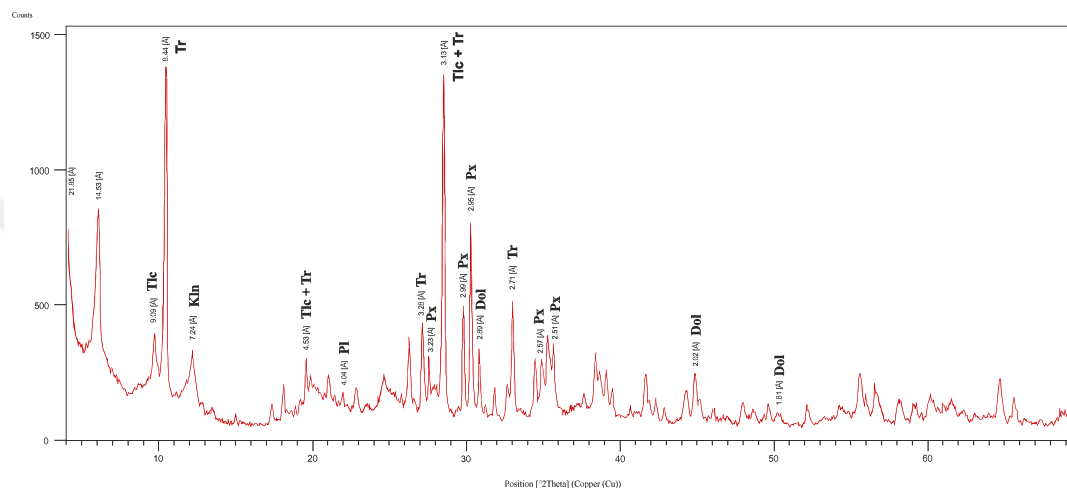


Figure 4.15. The bulk XRD diffractogram of main additive sample (Dol: Dolomite; Pl: Plagioclase; Px: Pyroxene; Tr: Tremolite; Tlc: Talc).

In the clay fraction analysis of the main additive sample (Figure 4.16.), 14 Å reflection does not show a total swelling but an intensity decreases along with a slight broadening. Upon thermal loading, it collapses towards 11 Å. This pattern coincides mostly with vermiculite. The 7 Å peak is stable under EG but totally collapses by heating to 550°C referring to the presence of kaolinite. Non-clay components of the bulk compositions stay stable under EG and heating to 350°C and 550°C.

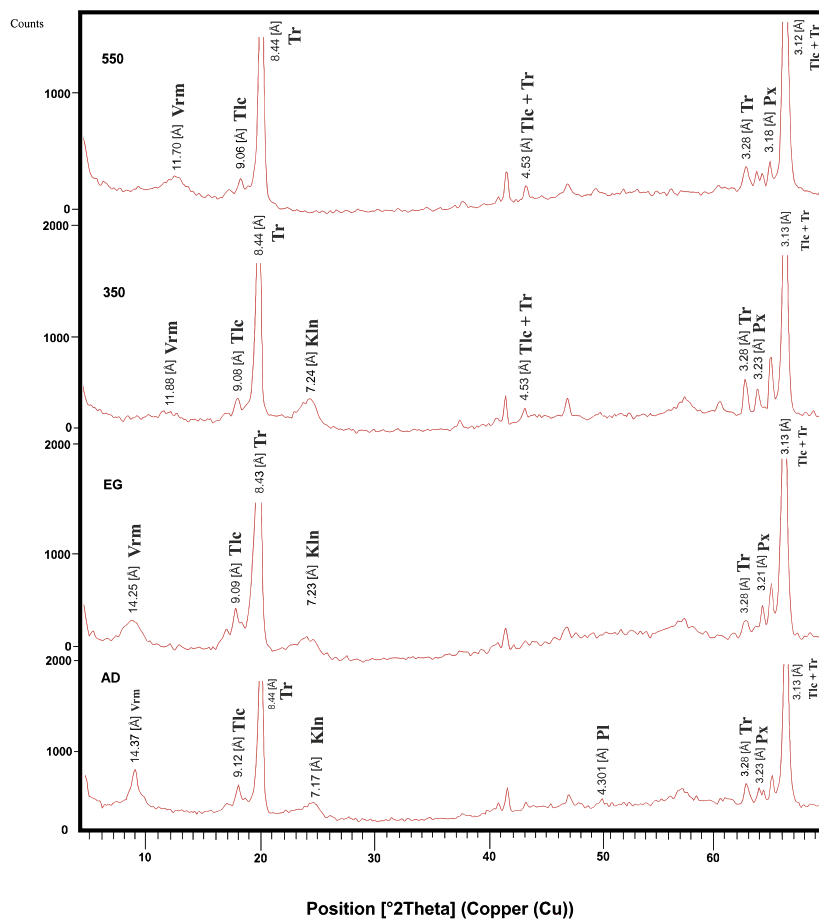


Figure 4.16. XRD diffractogram of clay fraction of main additive sample (Px: Pyroxene; Tr: Tremolite; Tlc: Talc; Pl: Plagioclase; Vrm: Vermiculite).

Figure 4.17. presents the diffractograms of the main additive sample heated from 400°C to 1000°C. At 400°C heating, there is no remarkable difference between bulk and heated diffractograms. However, at 800°C, dehydroxylation strongly removes the hydroxyls revealed by the collapse of 14 Å to 11 Å. Upon heating the sample to 1000°C, a new crystal is formed, defined by the reflection at 4.11Å as cristobalite.

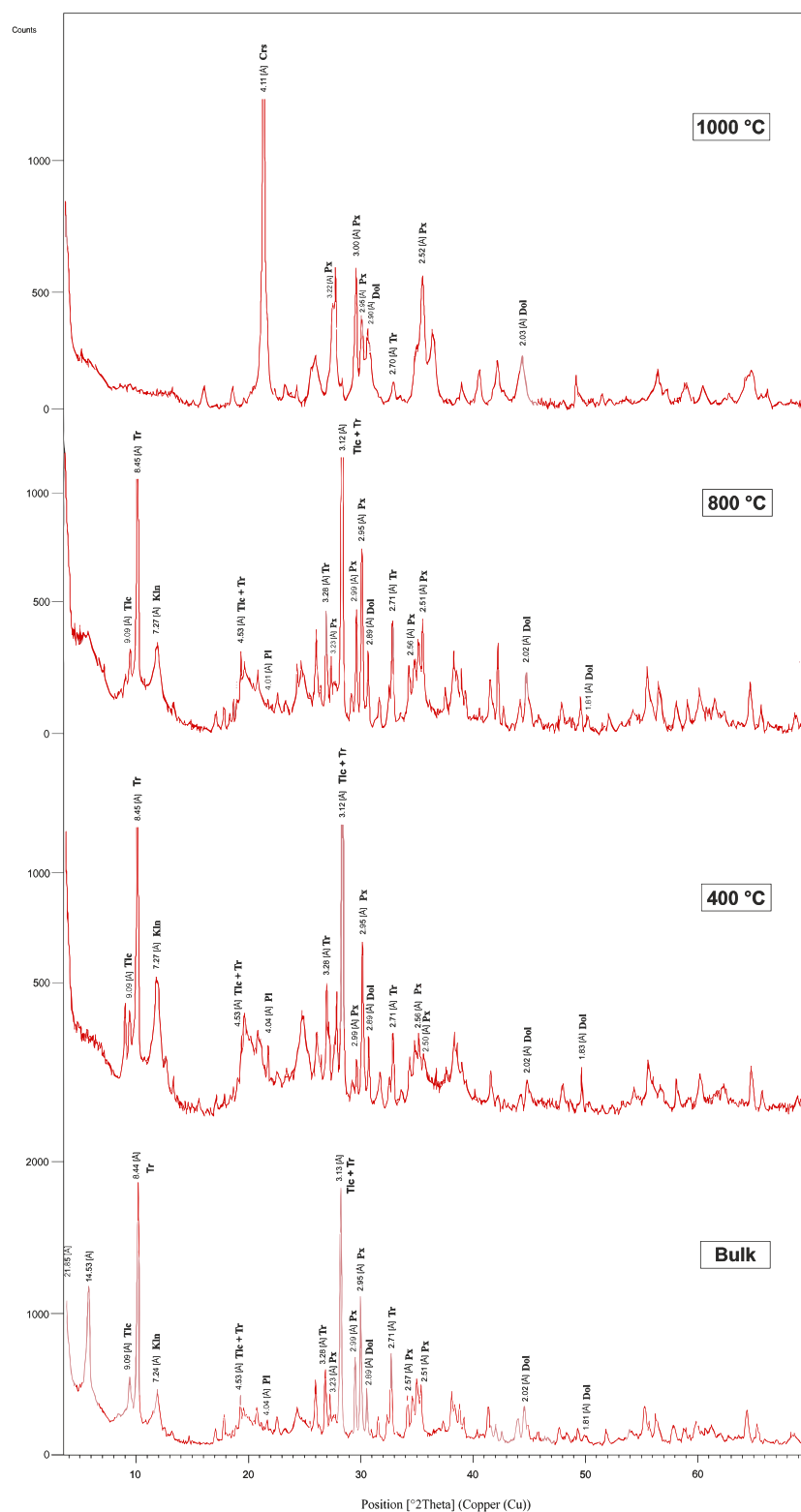


Figure 4.17. XRD Heated Bulk Result of Main Additive Sample (Vrm: Vermiculite; Dol: Dolomite; Pl: Plagioclase; Px: Pyroxene; Tr: Tremolite; Tlc: Talc; Crs: Cristobalite).

From the FTIR analysis of the main additive sample in Figure 4.18, clear spectral bands with respect to molecular structure related features can be drawn. Specifically,

the bands existing at 3691.14 cm^{-1} , 3618.01 cm^{-1} , and 3391.20 cm^{-1} could be attributed to the stretching vibrations of basically free and hydrogen-bonded hydroxyl groups (Madejová, 2003). Their existence hence proves that there must be correlated water molecules bound within the sample. Corresponding to this, an intense band located at about 1637.88 cm^{-1} was found within the spectrum in which this band may belong to the deformation vibrations of water molecules. So, that further proves that the sample is wet in structure or contains moisture content present within it. Further down, at lower wavenumbers, attention is attracted to a couple of bands: one appears at 995.58 cm^{-1} and the other at 908.83 cm^{-1} which are attributed to the bending of Al_2OH of kaolinite vibrations (Madejová, 2003; Jozanikohan and Abarghoeei, 2022). Further, these bands may also infer the existence of silica or silicate structures into the sample. If silicate structures exist, the band at 908.83 cm^{-1} could also relate to the mineral kaolinite (Si-O-Al). At still lower wavenumbers in the regions, one finds bands at 751.38 cm^{-1} , 639.98 cm^{-1} , 525.96 cm^{-1} , and 410.91 cm^{-1} that correspond to such feldspar vibrations (Farmer, 2000). Among them, metal-oxides indicate that there may be some oxidized metallic compound or mineral content in the sample. The hydroxyl groups indicate the entrapment of water or compounds having a hydroxyl group.

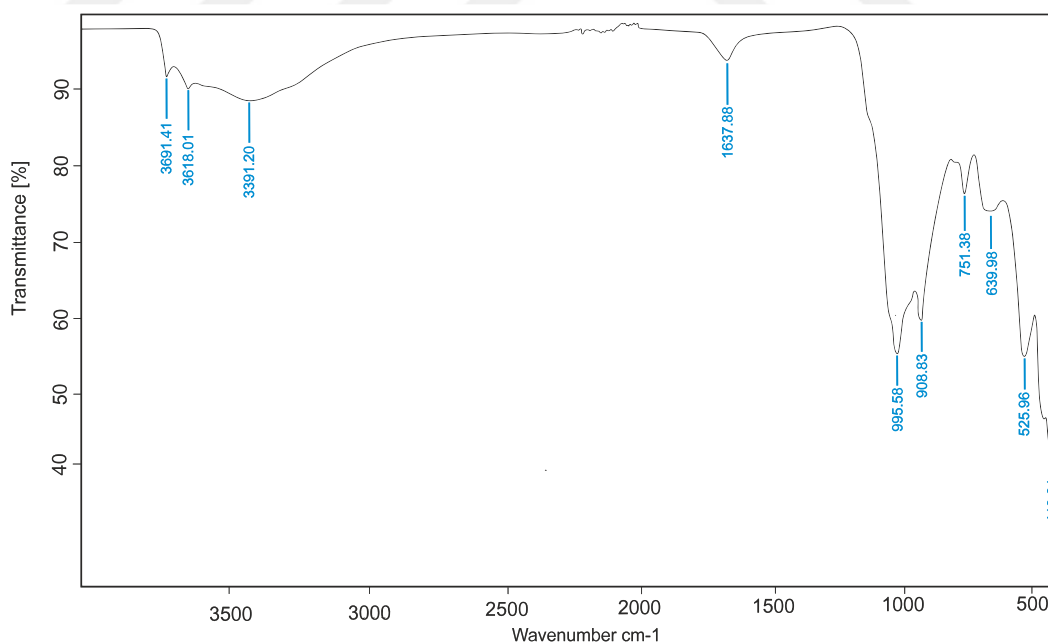


Figure 4.18. FTIR Spectrum of Main Additive Sample (DC2022L02-AM01)

4.3.1.2. Mineralogy of decoration material (DC2022L1-O1)

The most prominent and highly intense peaks correspond to the tremolite mineral, specifically at 8.41Å, 4.51Å, 3.38Å, 3.27Å, 3.12Å, and 2.70Å. The 2.95Å reflection refers to pyroxene. Plagioclase mineral peaks were observed at 4.02Å, 4.21Å, and 2.94Å, while calcite mineral peaks were found at 3.02Å and 2.30Å (Figure 4.19.). The decoration sample stands out for its high amphibole content (64%) and very low calcite, dolomite and kaolinite (total <10%).

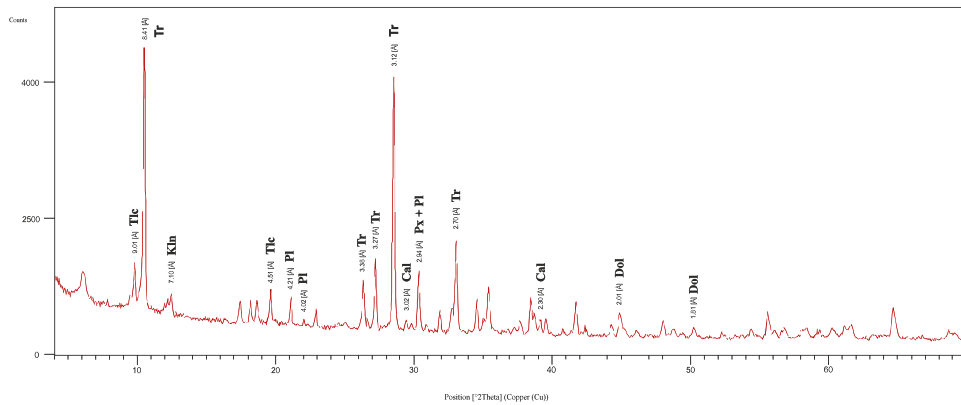


Figure 4.19. Diffractogram of bulk composition of Decoration Material (Cal: Calcite; Dol: Dolomite; Pl: Plagioclase; Px: Pyroxene; Tr: Tremolite; Kln: Kaolinite)

4.3.2. Geochemistry of additives

The major oxide composition of the main additive material-(DC2022L02-AM01) is given in Table 4.2. Quite similar to the Dont Clay sample, SiO₂ content of the main additive sample is high (49%) while Al₂O₃ is relatively lower (12.4%) clearly pointing to a low proportion of kaolinite in the bulk composition (Dondi et al., 2001). Total content of alkaline earth elements is higher than Al₂O₃; CaO is 11.4% and MgO is 12.4% regarding the Mg-bearing silicates, calcite and tremolite in the bulk composition. Alkaline elements, on the other hand, are very low in the bulk composition: K₂O <0.1; Na₂O 0.2%. Fe₂O₃ composition is 8.8% directly related with the pyroxene. LOI value is 5.35% which is in association with the bulk mineralogy, loss of hydroxyls and carbonates at high temperature.

Table 4.2. Major oxide compositions of main additive sample.

Sample ID	Main additive material (DC2022L02-AM01)
LOI (%)	5.35
Al ₂ O ₃ (%)	12.4
CaO (%)	11.4
Fe ₂ O ₃ (%)	8.8
K ₂ O (%)	< 0.1
MgO (%)	12.4
MnO (%)	0.2
Na ₂ O (%)	0.2
P ₂ O ₅ (%)	< 0.1
SiO ₂ (%)	49.0
TiO ₂ (%)	0.1

4.3.3. Thermal analysis of additives

Upon examining the DTA-TG thermogram of the main additive sample (DC2022L02-AM01), an initial and significant mass loss is observed on the TG curve at approximately 109.815°C. This mass loss is attributed to the dehydration process, where free or physically adsorbed water within the sample is removed. As the analysis progresses, a second mass loss occurs by endothermic reaction around 513.838°C and 574.838°C (Figure 4.20.). This temperature range is associated with the removal of structural water, specifically hydroxyl groups, from the clay minerals. This dehydroxylation process is represented by a distinct endothermic peak on the DTA curve. The occurrence of dehydroxylation in this temperature range indicates a significant disruption of the mineral structure and a transition towards an amorphous state. Additionally, very shallow and shallow endotherms are recorded at 861.107°C and 999.227°C, respectively pointing to the last stage of the dehydroxylation. At higher temperatures, around 1000°C, the DTA curve shows a pronounced exothermic peak. This reaction suggests the recrystallization of the sample, indicating a transformation into a more stable crystalline structure at elevated temperatures. Throughout the

analysis, a total mass loss of 8.0 mg is recorded, which can be attributed to the removal of water and other volatile components from the sample.

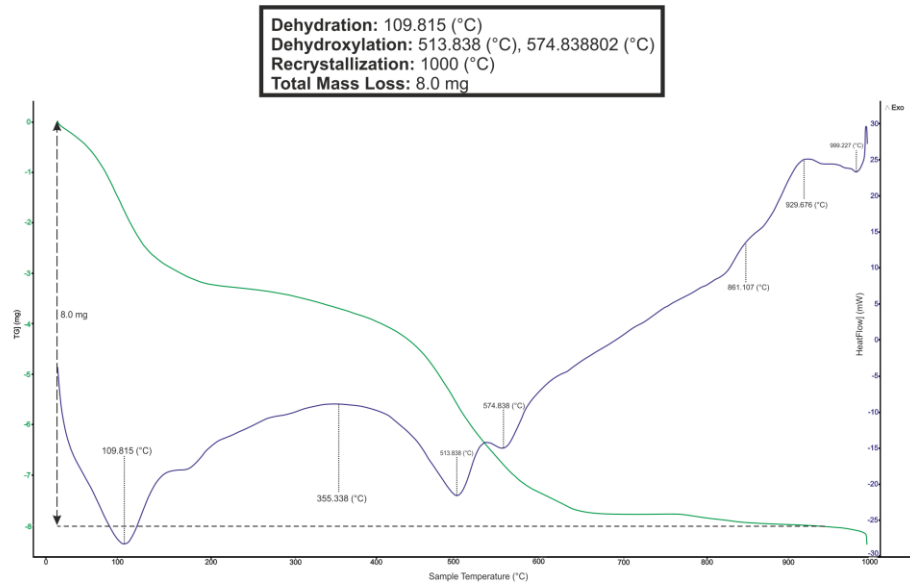


Figure 4.20. DTA-TG graph of Main Additive Sample (DC2022L02-AM01)

4.4. Characterization of Ceramic Muds

4.4.1. Mineralogy of ceramic muds

4.4.1.1. Mineralogy of 2021 Dont ceramic mud – MM1 (DC2022L1-MM1)

As explained in the methodology chapter, ceramic mud is the mixture of the main raw material and the main additive in a 5:1 ratio. Therefore, the diffractogram reveals the minerals of both samples. Based on the d-spacing values derived from the diffraction pattern, the peaks observed at 9.46 Å, 4.5 Å, 3.12 Å, 2.63 Å and 2.49 Å are attributed to the talc. Tremolite (amphibole) is another significant component of the sample with prominent peaks at 8.43 Å, 3.38 Å, 3.28 Å, and 2.71 Å. In the bulk diffraction of the sample, apart from talc and tremolite, the clearest, sharp and intense peaks at 3.34 Å and 3.04 Å belong to quartz and calcite. Pyroxene shows up at 2.95 Å, 2.58 Å, 3.23 Å and 2.99 Å reflections (Figure 4.21.).

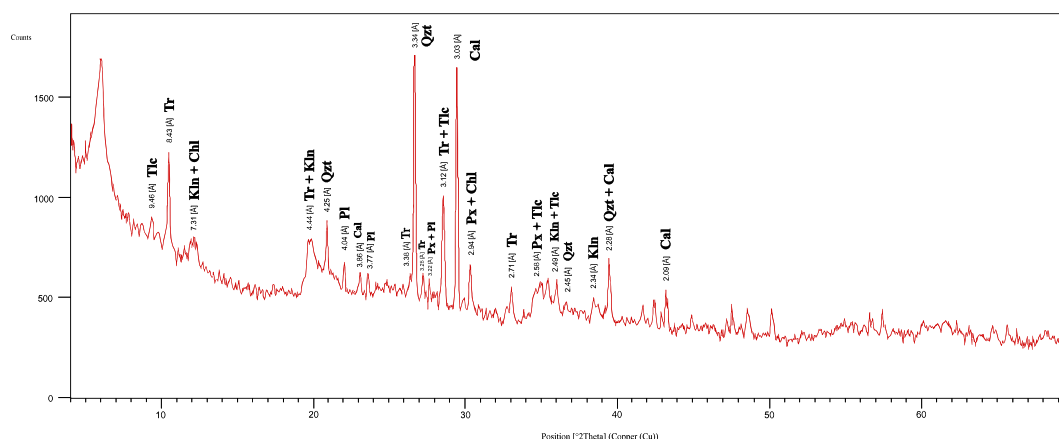


Figure 4.21. XRD Bulk Result of Ceramic Mud MM1 Sample (DC2022L1-MM1)

The FTIR analysis results of MM1 sample give significant information about the mineralogical composition of the sample. At 3382.47 cm^{-1} , a broad peak is observed, indicating that the sample has water molecules or hydroxyl groups; this band is generally assigned to water-containing structures of minerals such as clay minerals (Madejová, 2003; Jozanikohan and Abarghooei, 2022). Prior to this band, another peak was visible in the range from 3600 cm^{-1} to 3630 cm^{-1} in the linear graph, but this reading did not show any value for that. Probably, this should relate to the magnesium constituent of the sample giving hints to talc mineral within the composition (Ersoy et al., 2013). The band at 1638.09 cm^{-1} refers bending vibrations of H-O-H bound with water molecules thereby affirming the presence of water in the sample (Madejová, 2003; Jozanikohan and Abarghooei, 2022). Carbonates are indicated by peaks at 1414.37 cm^{-1} , which is the general frequency for carbonate minerals in the spectrum of CaCO_3 as well (Reig et al., 2002). Si-O bonds are indicated by prominent peaks at 992.01 cm^{-1} and 909.85 cm^{-1} , which again suggest that quartz or some other silicate minerals could have been the source of these vibrations. Vibrational modes of silicate minerals are represented by bands at 752.06 cm^{-1} and 675.92 cm^{-1} , showing clay minerals to be present. Lower frequency peaks such as these two are around 518.12 cm^{-1} and 422.87 cm^{-1} . Such peaks are characteristic of metal oxides (Farmer, 2002). These features thus inform us of probable bonds with metal oxide like Fe-O or Mg-O. From the FTIR spectrum, it can be said that there are silicate minerals, most probably quartz and clay minerals, and carbonates as well incorporation of water or hydroxyl groups in the MM1 sample. All these findings enhance the information regarding the mineral structure of the sample under discussion (Figure 4.22.).

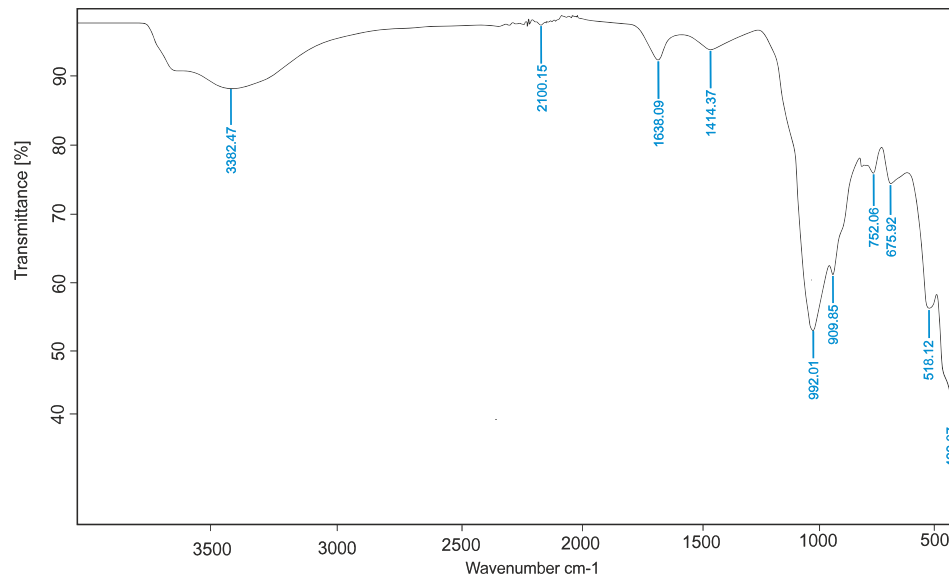


Figure 4.22. FTIR Spectrum of Ceramic Mud MM1 (DC2022L1-MM1)

4.4.1.2. Mineralogy of 2022 Dont ceramic mud – MM2 (DC2022L02-MM2)

The other ceramic mud sample is created by the local potter by mixing the main raw material and the main additive in a 5:1 ratio. Quartz and calcite with their prominent peaks at 3.34Å and 3.04Å appear in the diffractogram. Weak reflections of amphibole (tremolite) are determined at 8.4Å, 2.72 and 3.10Å. In addition to these, pyroxene appears at 2.95Å and 2.58Å reflections. With respect to the ceramic mud sample (DC2022L1-MM1), DC2022L02-MM2 has much higher proportions of well crystallized quartz and calcite in the mixture (Figure 4.23.).

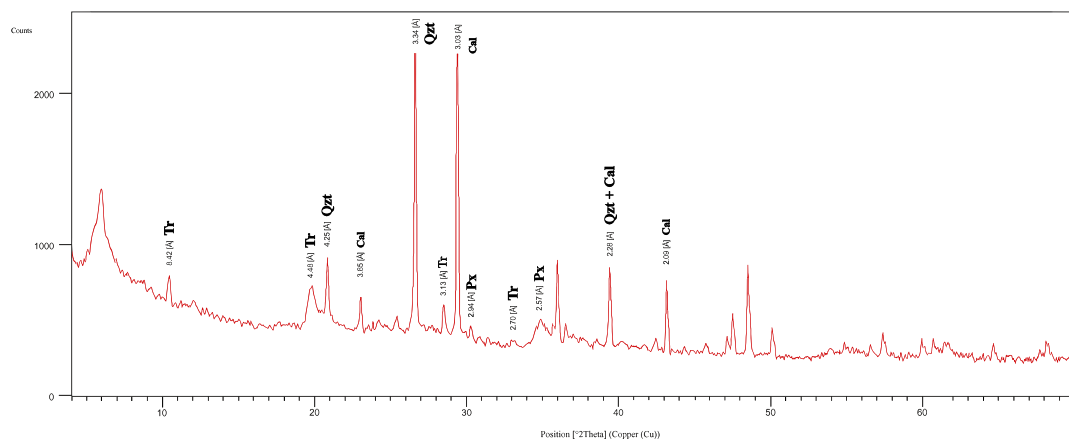


Figure 4.23. XRD Bulk Result of Ceramic Mud MM2 Sample (DC2022L02-MM2)

FTIR spectrum for the MM2 sample was very close to those identified in the MM1 sample as expected. A band situated in between 3600 cm^{-1} and 3630 cm^{-1} has been found as in MM1 suggesting the presence of magnesium and O-H (Jozanikohan and Abarghooei, 2022). The broad peak at 3380.05 cm^{-1} is characteristic of water or of hydroxyls bound to metal ions; the later would be expected in a hydroxyl-rich phase such as clay minerals. A further confirmation that the sample was wet comes from the band at about 1635.95 cm^{-1} that is due to the H-O-H bending vibrations of water: water is present in this sample. The peak at 1411.91 cm^{-1} is definitely carbonate minerals that have absorbed atmospheric CO_2 and specifically calcium carbonate (CaCO_3) (Reig et al., 2002). The Si-O bicornual absorption band, which was observed at 985.62 cm^{-1} and 872.67 cm^{-1} , was further confirmation of the existence of silicate minerals predominantly quartz and clay minerals. The bands at 777.18 cm^{-1} , 768.23 cm^{-1} , and 687.67 cm^{-1} are therefore assigned to the characteristic vibrational modes of silicate minerals, which confirm further the crystalline silicates (Madejová, 2003; Jozanikohan and Abarghooei, 2022). The low-frequency peaks at 504.31 cm^{-1} and 422.87 cm^{-1} are typically associated with metal oxides, showing the presence of bonds related to FeO or MgO (Figure 4.24.) i.e., iron oxide or magnesium oxide (Farmer, 2002).

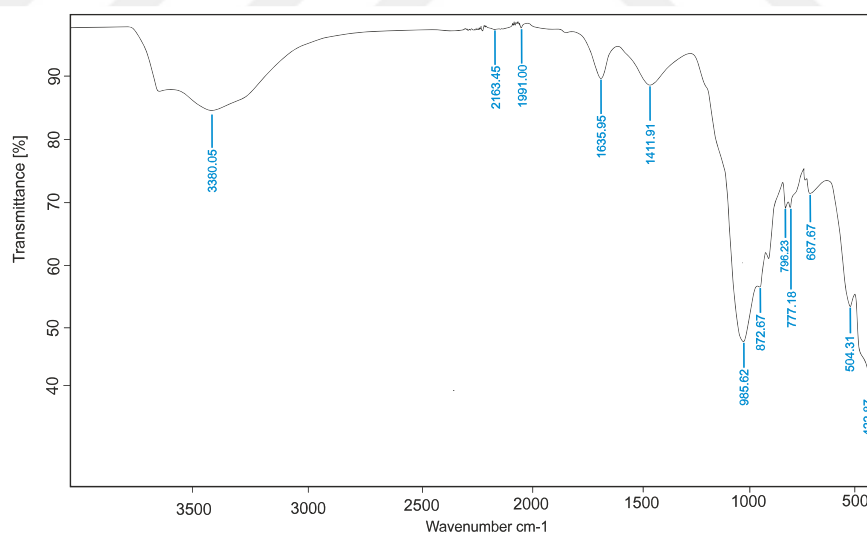


Figure 4.24. FTIR Spectrum of Ceramic Mud MM2 (DC2022L02-MM2)

The ceramic mud samples MM1 and MM2 exhibit a diverse mineral profile. MM1 demonstrates a notable presence of calcite (26%) with a balanced distribution of other minerals, whereas MM2 shows a similar composition but with a higher calcite content

(49%). This indicates a carbonate-rich composition, suggesting their potential suitability for specific industrial applications.

4.4.2. Geochemistry of ceramics muds

XRF analysis was conducted for the mixtures of the main raw material and additive. The results indicate similarly high values of silicium oxide and relatively low aluminum oxide. The MM1 sample has higher values of magnesium oxide, aluminum oxide, and iron oxide compared to the MM2 sample. Notably, the magnesium oxide content is 3.2% higher. In contrast, the calcium oxide content in the MM1 sample is 3% lower. The loss on ignition (LOI) ratio of the MM2 sample is higher than that of the MM1 sample. The higher CaO content in the MM2 sample could explain this difference in LOI ratios. Additionally, it is likely that the hydroxyl content of these mixtures, produced at different times, also varies. Another noteworthy observation is that the K₂O content is higher in the MM2 sample (Table 4.3.). Although the samples were collected from nearby locations, their chemical compositions may vary due to factors such as hydrogeological activities, climatic conditions, and organic matter content. The naturally fluctuating chemical content may also undergo proportional changes over time as a result of human influences, such as agricultural and mining activities.

Table 4.3. Major oxide compositions of ceramic muds.

Sample ID	Ceramic Mud MM1 (DC2022L1-MM1)	Ceramic Mud MM2 (DC2022L02-MM2)
LOI (%)	11.55	16.20
Al ₂ O ₃ (%)	12.3	10.7
CaO (%)	12.0	15.0
Fe ₂ O ₃ (%)	7.8	6.8
K ₂ O (%)	0.8	1.4
MgO (%)	7.5	4.3
MnO (%)	0.2	0.2
Na ₂ O (%)	0.4	0.2
P ₂ O ₅ (%)	< 0.1	0.1

Table 4.3. (continue)

Sample ID	Ceramic Mud MM1 (DC2022L1-MM1)	Ceramic Mud MM2 (DC2022L02-MM2)
SiO ₂ (%)	46.8	44.33
TiO ₂ (%)	0.4	0.5

4.4.3. Thermal analysis of ceramic muds

In the DTA-TG analysis of the MM1 sample, the first notable event is the endothermic reflection observed at 110.483 °C, which indicates the dehydration process where the sample loses adsorbed or free water molecules (Figure 4.25.). This process is commonly observed in water-containing minerals and is particularly associated with structures such as clay minerals. As the temperature increases, two distinct dehydroxylation events occur: the first at 483.241 °C and the second at 802 °C. The dehydroxylation at 483.241 °C is typically linked to the loss of structural hydroxyl groups in minerals, such as clays, that contain these groups. The second dehydroxylation event at 802 °C is associated with the removal of more stable hydroxyl groups, often related to complex silicate minerals. One of the most significant events in the sample's thermal behavior is the recrystallization process, which occurs at 1000 °C. The pronounced exothermic reaction observed at this temperature suggests that the mineral structures in the sample undergo recrystallization, leading to a more ordered crystalline form. A total mass loss of 11.3 mg was observed, which is attributed to the thermal removal of water, hydroxyl groups, and other volatile components. The remarkable point on this thermogram is the deepening of third endothermic peak as the materials are mixed and the silicate and carbonate amounts increase.

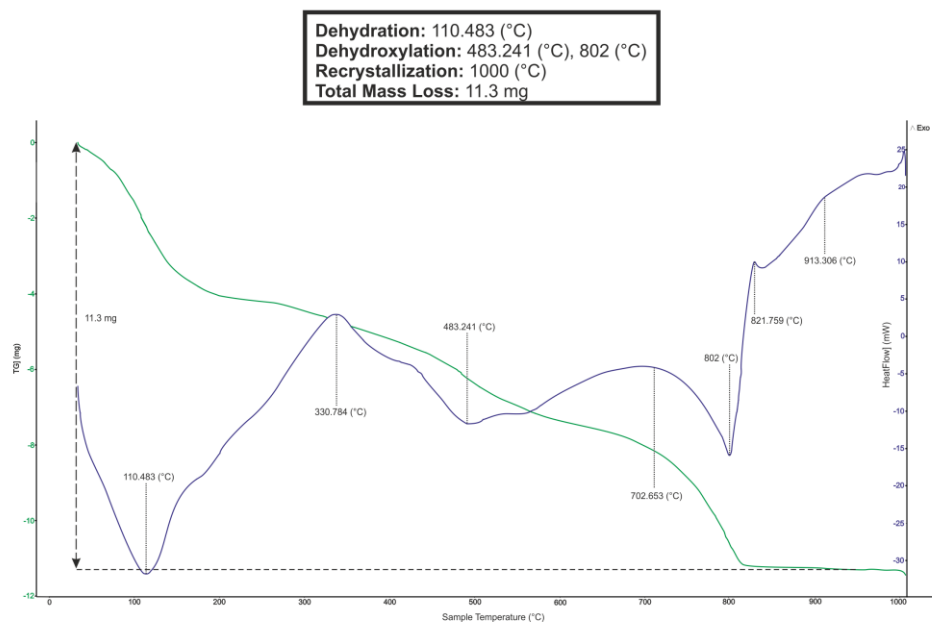


Figure 4.25. DTA-TG graph of Ceramic Mud MM1 Sample (DC2022L1-MM1)

In the DTA-TG analysis of the MM2 sample (Figure 4.26.), the endothermic reaction observed at 119.104 °C indicates the dehydration process, where the sample loses adsorbed or free water molecules. This process is a common thermal event, particularly associated with water-containing structures such as clay minerals. As the temperature increases, relatively shallow endotherms at 560°C and much deeper at 835.57 °C are observed that are directly related with the dehydroxylation process. At this temperature, the removal of structural hydroxyl groups from the sample occurs. The exothermic reaction observed at 1000 °C corresponds to the recrystallization process. The pronounced exothermic reaction at this temperature indicates that the mineral structures in the sample undergo recrystallization, resulting in a more ordered crystalline structure. Throughout the analysis, the sample experiences a total mass loss of 17.0 mg. This mass loss is due to the thermal removal of water molecules, structural hydroxyl groups, and other volatile components.

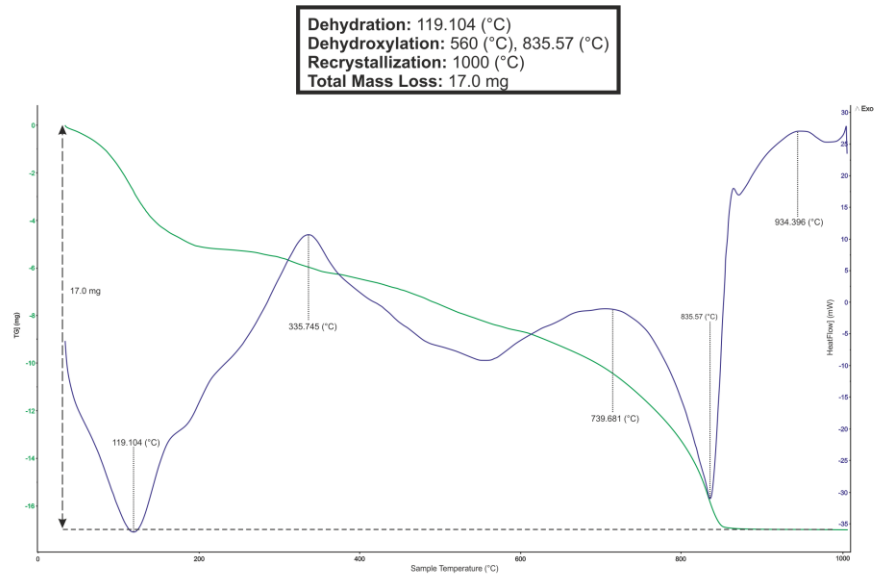


Figure 4.26. DTA-TG graph of Ceramic Mud MM2 Sample (DC2022L02-MM2)

5. DISCUSSION

5.1. Evaluation of The Results of Multi -Analytical Characterization

This section begins by comparing two main raw materials, referred to as Dont clays from Dont plateau (DC2022L02-M01) and Esenköy village (DC2022L09-ES). These two materials are quite similar in terms of their mineral compositions. Both clay materials contain talc, tremolite, pyroxene, plagioclase, and kaolinite. The Dont clay exhibits swelling behaviour of vermiculite and collapses to 10 Å upon thermal loading, while in the case of Esenköy clay, the presence of mixed-layer clays is noted, and swelling properties are observed when it is saturated with ethylene glycol. According to semi-quantitative analysis, Dont clay has higher contents of tremolite (27%) and pyroxene (36%), whereas Esenköy clay has higher contents of smectite (16%) and kaolinite (18%). When comparing the reactions of crystal phases to different temperatures, it has been found that neither the Dont clay sample nor the Esenköy sample complete the dehydroxylation phase up to 900 °C. Consequently, it has been determined that all hydroxyl groups collapse between 800 and 900 °C. Both samples similarly exhibit the development of a new crystal phase around 1000 °C with d-values of 4.08-4.11 Å. Thermal stabilities of these two raw materials are established on four-three major endothermic reactions respectively related with dehydration and dehydroxylation reactions and an exothermic reaction at about 1000 °C.

For the production of ceramic mud, 1 unit of additive material is mixed with 5 units of raw material. The comparison of additive material with the main raw materials reveals very similar mineral compositions along with reflections of vermiculite. XRD analyses of the heated additive materials also show the development of a new phase with a d-value of 4.11 Å in the additive material. Two ceramic mud samples created by using the same recipe at different times are also compared with each other. Muds are MM1 sample (DC2022L1-MM1) prepared in 2021 and MM2 sample (DC2022L02-MM2) prepared in 2022. The distinct differences of these mixtures are related with the

abundance of the minerals (Table 5.1.) and both ceramic muds are enriched in calcite (26% for MM1 and 49% for MM2).

Table 5.1. Mineral content of ceramic mud samples.

Sample	Ceramic Mud MM1	Ceramic Mud MM2
Talc (%)	4	2
Pyroxene (%)	16	7
Plagioclase (%)	3	1
Quartz (%)	18	30
Amphibole (%)	19	8
Calcite (%)	26	49
Dolomite (%)	0	0
Kaolinite (%)	13	3
Smectite (%)	0	0
Hematite (%)	1	0

This finding is also in coherence with the CaO values of the samples. Geochemically, the alkaline earth element compositions of both ceramic muds are equal (19.5%), as are their thermal stabilities with definite endotherms at almost same temperatures.

Comparison of the major oxide compositions and LOI values of all samples shows that Dont clay and Esenköy clay samples are enriched in SiO₂, while Al₂O₃ is relatively lower. Dont clay sample is enriched in alkaline earth elements (23.2% in total) and deficient in alkaline elements. The Esenköy clay sample, with respect to Dont clay, has 10.9% alkaline earth in the bulk composition. The potential reason for the difference in alkaline earth element levels could be the MgO content in the samples. Higher contents of tremolite, pyroxene and the presence of dolomite in the Dont clay sample result in a higher MgO concentration, thereby making it richer in alkaline earth elements. The Esenköy sample is enriched in Fe₂O₃ (19.4%) quite consistent with the Fe-bearing mineral abundance in the bulk composition. In addition, LOI value (10.15%) is higher than that of Dont clay (6.45%).

The main additive material provides high alkaline earth composition to the ceramic mud samples (prepared with Dont clay) and make the mud more suitable for ceramics.

Talc in both raw materials provide Mg and Si that triggers the formation of Mg silicates and making the ceramic body or the pottery much stronger and dense. Especially the decomposition of talc at around 870-890 °C releases the free silica that would end up a formation of new crystals (Sallam et al., 1984). The most notable difference in the comparison of the additive material used for decoration with other samples is the relatively high amphibole content (64%).

The presence of Mg-bearing minerals in both raw materials strongly affect the mechanical strength of the pottery. Sallam et al. (1984) experienced that addition of talc to a ceramic mud and monitored the dehydroxylation at 960°C and crystallization of cristobalite or enstatite above 1000 °C. Similarly, the study of Il'ina and Lebedeva (2012) shows the loss of structural water from talc and tremolite at 930-980 °C. Addition of talc and tremolite as fillers to the ceramic mud strongly influence the durability and strength of the ceramic body with respect to the addition of feldspar Il'ina and Lebedeva (2010).

Carbonates are also the other important components of the ceramic bodies, especially for the preparation of porous media due to the decomposition of carbonates into CaO and CO₂ during sintering (Emani et al., 2014). Additionally, addition of calcium carbonate increases the mechanical strength to moisture expansion and reduces the firing shrinkage; while magnesium carbonate increases the moisture expansion (Lira et al., 1990).

A comparison was made between the mineralogical analysis data of samples obtained from the Dont plateau (Dont clay, Esenköy sample, main additive sample and ceramic muds) and several reference clay products. In this context, the samples were plotted on the ternary diagram of Fabbri and Fiori (1985). The study by Fabbri and Fiori (1985) evaluated high-quality industrial clays that are globally recognized as reference materials. Accordingly, the ternary diagram proposed by Fabbri and Fiori (1985) was utilized in this study to compare the analyzed samples with these reference clays. The plotting the Dont samples (Dont clay, Esenköy sample, main additive material and ceramic muds) on the ternary diagram the corners of which are occupied with (Fe₂O₃+CaO+MgO+TiO₂+Na₂O+K₂O)-SiO₂-Al₂O₃, reveals that all the samples do not fall in the red stoneware, structural ceramic, or white stoneware fields (Figure 5.1.).

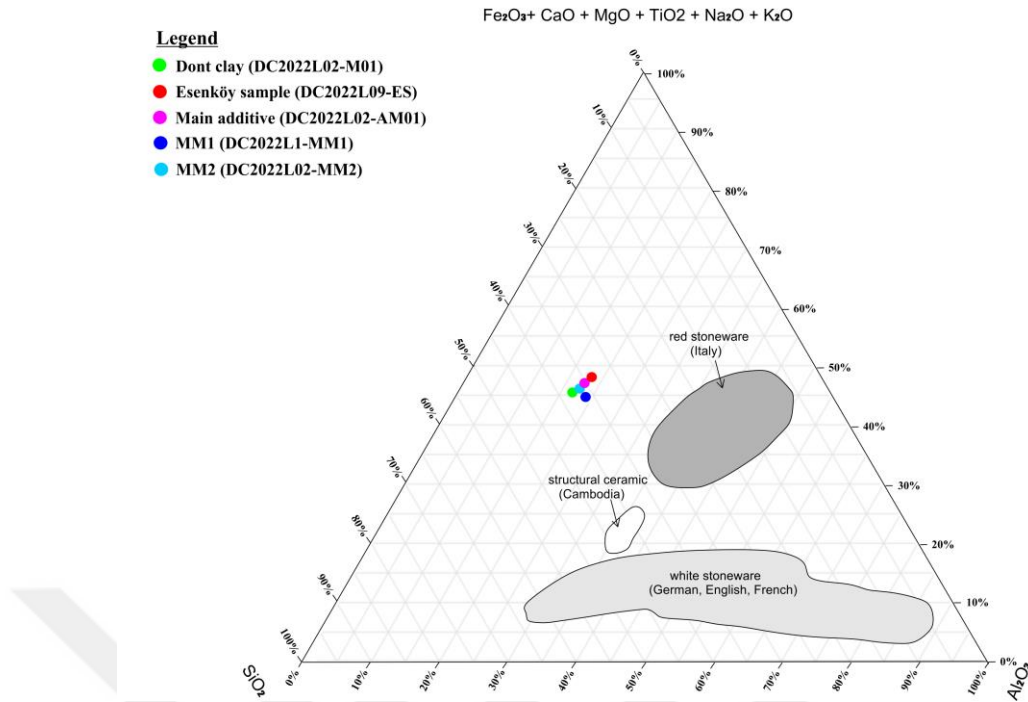


Figure 5.1. Triangular diagram of clay-rich materials of Fabbri and Fiori (1985) with Dont samples

In another evaluation, the Dont samples were compared with the reference industrial applications bordered in the ternary diagram of Strazzera et al. (1997). The Strazzera ternary diagram is a commonly utilized reference tool in the ceramics industry, employed to evaluate the potential application areas of ceramic raw materials in terms of production suitability. The findings indicate that raw material samples of the Dont clay material (Dont clay and Esenköy sample) do not individually fall within the structural clay products or clay roofing tiles fields (Figure 5.2.). However, the main additive material and ceramic muds are positioned within the clay roofing tiles field. This suggests that Dont clay, on its own, is insufficient for such industrial applications but becomes a more suitable material when prepared with an additive material and transformed into ceramic mud (Figure 5.2.). Furthermore, the MM1 ceramic mud is located very close to the structural clay products field, unlike the MM2 ceramic mud. The potential reasons for the differences observed between the MM1 and MM2 samples may include microenvironmental variations during raw material sampling, the amount of organic matter influencing carbonate content, or changes in precipitation patterns affecting the deposition of clay minerals. Such factors are likely attributable to the fact that the sampling process was conducted at different times.

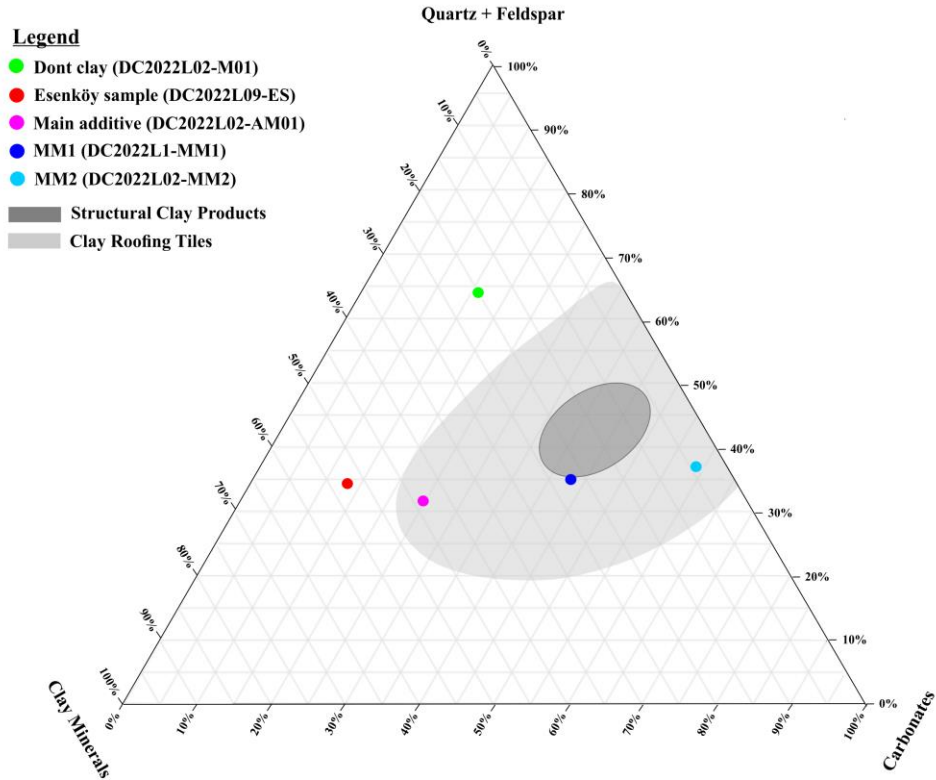


Figure 5.2. Strazzera ternary diagram created by taking into account geochemical data of Dont plateau Strazzera et al. (1997)

5.2. Multi Analytical Comparison of Dont Plateau Samples And Reference Clays

The Dont clay samples have been compared with selected studies that include similar lithologies and applied similar methodologies. Ukrainian clay (UKC) as globally one of the most utilized reference clay for ceramics (from Agayev and Küçükuysal, 2018; clay material UKC) and the clay material from Algeria (from Abdelmalek et al., 2017; clay materials E01, E02, E03) due to its lithological similarities to the Dont clay materials are selected for the comparison. Additionally, clay-rich materials (TR1, TR2, TR3) from Denizli region studied by Semiz (2017), Karacasu clay (KRC) from Aydın region studied by Özkan (2014) and Uşak clay (USC) from Uşak region studied by Agayev and Küçükuysal (2018) are included in this comparison as the clay materials from western Anatolia.

5.2.1. Mineralogical comparison

In the Dont raw material samples (Dont and Esenköy), minerals such as talc, pyroxene, amphibole, plagioclase, quartz, and kaolinite were identified. Additionally, calcite and smectite minerals were also observed in the Esenköy sample, while the presence of dolomite was noted in the Dont sample. Both samples contained small amounts of hematite. The mineralogical composition of the Denizli and Aydın clays from nearby regions generally includes quartz, illite, kaolinite, smectite, and hematite (Semiz, 2017). This reveals that TR1, TR2 and TR3 samples from Denizli region and KRC sample from Aydın region are mineralogically to the clay materials of this study. However, the presence of minerals like talc, amphibole, pyroxene, and plagioclase in the clay materials of this study creates a composition that does not fully align with the typical characteristics of clays from these regions. Ukrainian clay sample UKC and the clay samples from Algeria E01, E02, E03 consist of quartz, kaolinite, illite, and dolomite. Abdelmalek et al. (2017) also documents that samples E01, E02, E03 include plagioclase and calcite. E01, E02, Despite some similarities in composition, the most fundamental difference between Dont clay and other samples is the varying amounts of kaolinite as the primary clay mineral. According to the kaolinite values provided in Table 4, the Dont sample contains 7% kaolinite, while the Esenköy sample contains 18%. In the Denizli samples, these values range from 1% to 10%, and the kaolinite content of the reference Karacasu clay (KRC) is 22% (Semiz, 2017). Similarly, the kaolinite content of Uşak clay (USC) was calculated as 12% (Agayev and Küçükuysal, 2018). Based on these results, it can be stated that the clay materials from this region generally have low kaolinite values. The proportional differences among reference clays can be attributed to various factors, such as parent rock composition, climatic conditions, hydrochemical influences, and geological formation processes. In the Algerian samples, kaolinite contents range from 10% to 38% (Abdelmalek, 2017), while the Ukrainian clay (UKC) contains 51% kaolinite (Agayev and Küçükuysal, 2018), indicating that these two samples have significantly higher kaolinite contents.

5.2.2. Geochemical comparison

The chemical data of the samples in this study, along with reference samples, were plotted on a ternary diagram comparing industrial ceramic products with reference clay materials from Fabbri and Fiori (1985) (Figure 5.3.). According to this comparison three main regions are identified. The red stoneware region is characterized by its high iron oxide content, which gives the material a reddish or brownish hue upon firing. These materials represent ceramics fired at medium temperatures (1000–1200°C) and are typically used for decorative or everyday purposes. The white stoneware region, on the other hand, has a lower iron oxide content, resulting in lighter or white-colored materials. It is characterized by high silica and alumina content and is usually fired at higher temperatures (1200–1300°C). Due to its vitreous nature, it is commonly utilized in aesthetic or functional products. Finally, the structural ceramic region encompasses materials fired at lower temperatures (850–1100°C) with moderate silica and alumina content. This category includes porous construction materials such as bricks and tiles (Fabbri and Fiori, 1985).

The diagram shows that, the reference clays USC (Uşak clay), UKC (Ukrainian clay), and KRC (Karacasu clay) are positioned within or near the white stoneware field. This positioning is due to their enrichment in aluminum and silicate despite their low Fe_2O_3 and other oxide contents. Among the samples from the Dont plateau and reference clays, the only sample located in the red stoneware field is TR3, one of the Denizli samples. The Al_2O_3 content of the TR3 sample is 26.21%, which can be considered relatively high. In addition to its aluminum content, its high silica and Fe_2O_3 levels allowed it to be classified in the red stoneware field. In the structural ceramic products field, the Algerian sample E01 and the Aydın sample, Karacasu clay (KRC), can be included. The Al_2O_3 content of the E01 sample is 19.62%, while the KRC sample has an Al_2O_3 content of 17.31%. The high silica contents of these two samples, combined with their aluminum content, seem to significantly influence their positioning in the ternary diagram. The Dont samples (Dont clay, Esenköy sample, primary additive, and ceramic muds) are generally characterized by high silica and low aluminum content. Their relatively high Fe_2O_3 and other oxide levels allow them to be positioned closer to the red stoneware field. The TR1 sample from Denizli exhibits a composition similar to the Dont samples, with nearly identical aluminum content (Dont samples have an

average of 11.78% Al_2O_3 , while the TR1 sample contains 11.69% Al_2O_3). For this reason, TR1 is positioned very close to the Dont samples in the diagram (Figure 5.3.). The results indicate that aluminum content is a significant factor for all samples and that a high silica content alone is insufficient. In general, while some reference clays may be suitable for industrial ceramic applications, the current state of the samples from the Dont plateau renders them unsuitable for such uses. The primary reason for this is their low aluminum content. While the aluminum content influences the general suitability of the materials, other oxides in the composition affect their positioning within the diagram's fields. Making Dont clay suitable for industrial ceramic use depends on several conditions. For example, to be used in the red stoneware field, increasing the aluminum content alone might be sufficient. To achieve this, the aluminum level must reach approximately 30%, which would require an increase of more than 50%. To be used as structural ceramics or white stoneware, increasing the aluminum content must be accompanied by reducing Fe_2O_3 and other oxide levels. Among the reference clays, the Denizli (TR1, TR2, TR3) and Algerian clays (E01, E02, E03) yield results most similar to Dont clay. This similarity is largely due to the parent rocks from which they are derived, as they share similar lithological characteristics, resulting in comparable chemical compositions.

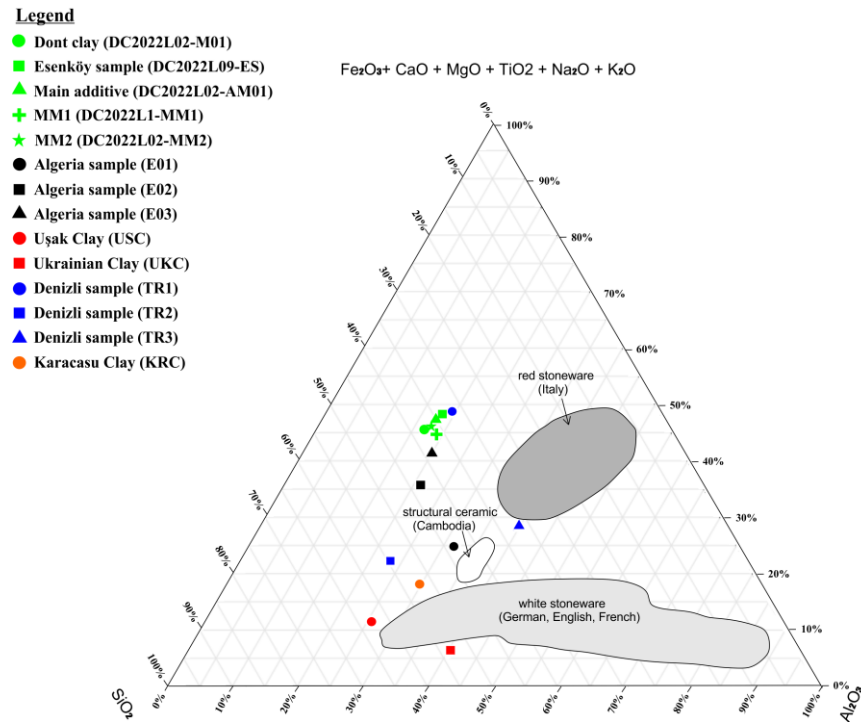


Figure 5.3. Triangular diagram of Fabbri and Fiori (1985) with Dont plateau samples and Reference Clays (Black colored plots; Algeria samples, red colored plots; Ukraine and Uşak clays, Blue colored plots; Denizli samples, orange colored plots; Karacasu clay).

In the ternary diagram (Clays-Carbonates-Quartz and Feldspar) proposed by Strazzera et al. (1997), samples from the Dont plateau and reference samples were plotted (Figure 5.4.). According to the ternary diagram, the Denizli samples (TR1, TR2, TR3), Karacasu clay (KRC), Ukrainian clay (UKC), and Uşak clay (USC) fall outside the application fields indicated in the diagram and are deemed unsuitable for these applications. The primary reason for this is their very low or absent carbonate content. Among other results, the Algerian samples E02 and E03 are positioned within the clay roofing tiles field. Additionally, the E03 sample is also close to the structural clay products field. The E01 sample, however, falls outside these fields due to its lower carbonate content compared to the other two samples. Among the Dont plateau samples, except for the raw materials (Dont clay and Esenköy sample), all other samples are positioned within the clay roofing tiles field. The MM1 sample is also suitable for structural clay products. The MM2 sample, despite having lower clay mineral content, is included in the applicable field due to its high carbonate and quartz-feldspar content. The Dont samples (Main additive, MM1, MM2) positioned within the application fields differ from the other reference samples primarily due to their high carbonate content. Thus, in this evaluation proposed by Strazzera et al. (1997), carbonate content has been the most significant factor determining the industrial applicability of clay materials in these fields. Additionally, as observed in the ternary diagram by Fabri and Fiori (1985), the ternary diagram by Strazzera et al. (1997) also shows that the Dont clay samples exhibit similarities to the Algeria samples.

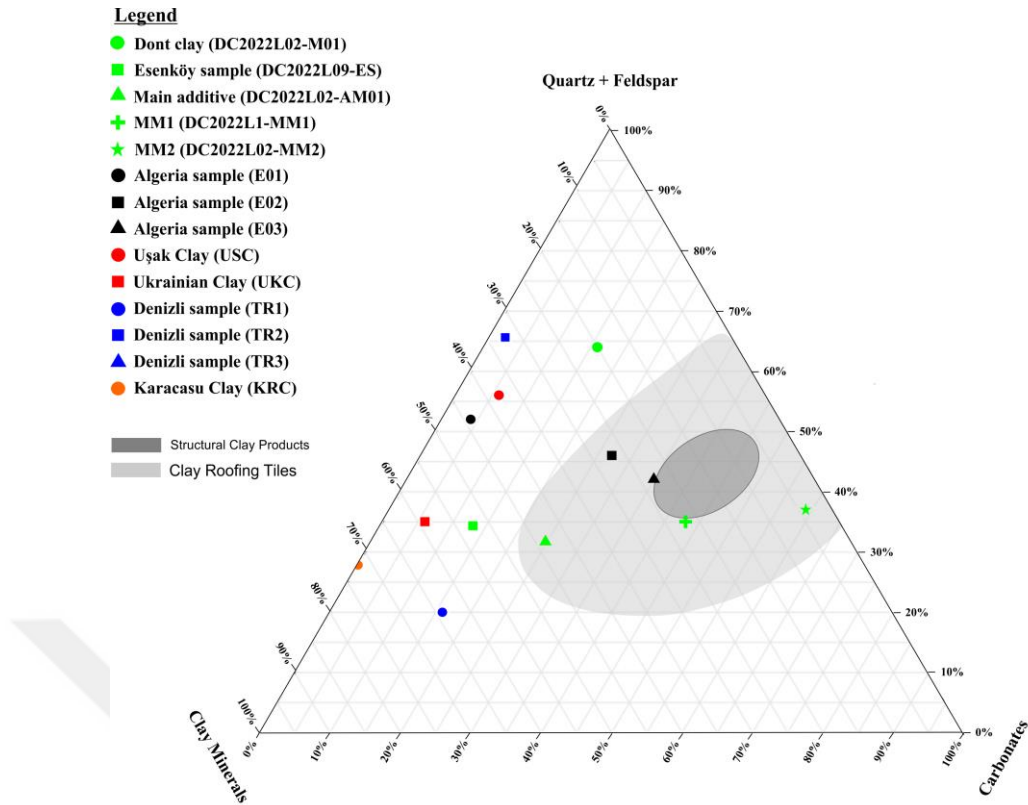


Figure 5.4. Strazzera et al. (1997) ternary diagram created with Dont plateau samples and Reference Samples Data (Black colored plots; Algeria samples, red colored plots; Ukraine and Uşak clays, Blue colored plots; Denizli samples, orange colored plots; Karacasu clay).

5.2.3. Thermal properties comparison

The DTA and TG curves were analyzed to compare the thermal properties of the samples taken from the Dont plateau and reference clays (Table 5.2.). In all samples, the first endothermic reaction occurred at approximately 100 °C, indicating the removal of absorbed water from the system (dehydration). The second endothermic reactions, associated with the dehydroxylation of hydroxyl groups (or kaolinite dehydroxylation), occurred around 450 °C in the Denizli samples (TR1, TR2, TR3). In the Uşak clay (USC), Ukrainian clay (UKC), Karacasu clay (KRC), and Dont clay samples, this temperature was approximately 500 °C. In the Esenköy sample and MM2 ceramic mud, the dehydroxylation temperature is approximately 60 °C higher compared to the other samples. In contrast, dehydroxylation in the MM1 ceramic mud occurs at 483 °C. The higher dehydroxylation temperature observed in the Esenköy and MM2 samples can be explained by the higher kaolinite and smectite contents. When examining total mass loss, it is observed that this rate is higher in the Denizli

and Dont samples compared to other reference samples. This may be related to the higher amount of absorbed water or interlayer water present in these samples.

Table 5.2. Thermal responses of she samples and the reference materials

Sample	Dehydration (°C)	1st Dehydroxylation (°C)	Total Mass Lost (%)
Dont Clay	111.1	501.8	8.9
Esenköy	125.8	565.2	18.4
Main Additive	109.8	513.8	23.7
MM1	110.4	483.2	16.1
MM2	119.1	560.0	20.8
USC	100.0	495.0	4.5
UKC	100.0	505.0	6.7
TR1	100.0	450.0	22.7
TR2	100.0	450.0	9.6
TR3	100.0	450.0	18.5
KRC	100.0	500.0	5.91

6. CONCLUSION

With the aim of characterization of the clay materials that are traditionally utilized for pottery making in Dont (Fethiye), multi-analytical approach was followed to determine the suitability of these materials for the industrial application in ceramics.

The following conclusions were drawn:

- Dont clay materials are the products of weathering and erosion and therefore represent weathering minerals of the mafic parent rocks as well as the terrestrial clastics in the region.
- The traditional pottery-making procedure in Dont is to use the fine fraction (less than 3mm) of the Dont clay and mix it with cream-colored fine-grained clay materials. Decorative figures were also emplaced with finders of the potter after firing the pots in a bonfire using juniper wood.
- Quartz, plagioclase, calcite, talc, tremolite and pyroxene are the non-clay minerals determined in the bulk composition of the raw materials of the Dont plateau (Dont clay and Esenköy sample). Smectite and kaolinite with minor vermiculite are the clay minerals of the fine fractions of the samples.
- The use of the main additive material in the clay prepared prior to ceramic production enhances the strength of the ceramic body due to its high silica content.
- The presence of talc in the Dont, Esenköy, and additive samples makes the resulting clay more suitable for ceramics by providing a high alkaline earth composition and triggering the formation of magnesium silicates, which strengthen the ceramic body and increasing the durability of the clay materials up to higher temperatures.
- Talc interacts rapidly with oil and, when combined with carbonates, transforms into an excellent fluid. These characteristics make talc significant for Dont clay.

- Dont clay is a material characterized with high silica and low aluminum oxide contents. Due to its low aluminum oxide content, it is unsuitable for use in structural ceramic products or high-quality stoneware.
- Dont clay, in its raw state, appears unsuitable for industrial applications in ceramics like redstone ware, structural ceramics or white stoneware ceramics. However, when prepared with an appropriate recipe forming like ceramic muds in this study using a primary additive material, the resulting ceramic clays become suitable for structural clay products and roofing tiles. This finding highlights the potential versatility of the ceramic clay for a broader range of applications.
- Another conclusive remark of this study is the detection of the possibility of primary additive material in industrial applications without requiring any additional mixing due to high silica and magnesium content with abundance of tremolite in the bulk composition.
- The thermal stabilities of the Dont and Esenköy samples are observed around 800°C, indicating that the resulting ceramic clay is suitable for firing at medium temperatures.

This thesis study I recommends further technological tests such as water absorption capacity, unconfined compression strength, plasticity index, and cation exchange capacity to investigate the possibility of Dont clay for potential industrial usage.

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