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ANALYTICAL DETERMINATION OF CLAY CONTENT IN MARMARA REGION SANDS USING GRAVIMETRIC, XRD AND FTIR METHODS

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Abstract
In this study, sand samples obtained from various suppliers in the Marmara Region were analytically evaluated to determine their clay content and mineralogical composition for potential use in construction chemical formulations. A gravimetric method was applied to separate quartz, feldspar and clay fractions, while phase identification and quantitative mineralogical analysis were performed using X-ray diffraction (XRD) supported by the Rietveld refinement method. The presence and relative amount of clay minerals were further assessed by Fourier transform infrared spectroscopy (FTIR), focusing on shifts in the Si–O stretching vibration near 1000 cm^{-1} and the Al–OH stretching band around 3600 cm^{-1}. The analyses showed that sand samples from Arnavutköy contained relatively low clay impurities, whereas Çerkezköy samples exhibited higher levels of clay and feldspar. The results demonstrate that the combined use of gravimetric analysis, XRD and FTIR provides a reliable analytical approach for determining low-level clay content in sand and supports the evaluation of raw material suitability for cement-based construction chemicals.

Keywords: Sand, clay, XRD, FTIR, cement-based construction chemicals

1. Introduction

The categories of cement-based construction chemical products include tile adhesive, joint filler, grout mortars, flooring products, water proofing and thermal insulation materials, and repair mortars. These materials comprise various types of binders and additives, depending on the desired properties [1,2]. With the curing effect of the hydration reaction that occurs when cement and water are combined, the used fillers reduce the spontaneous shrinkage and splitting of the cement paste to a certain extent [3]. In addition, the filler content affects the mixing time, setting time and condensation time. The particle size of the used filler, its composition (impurities, etc.), and the current moisture condition all influence the quantity of water needed to prepare the mixture. The physical and structural properties of the fillers vary according to the region where they are extracted, and the purification processes carried out by some manufacturers also affect the content [4–6].

Clays, which exist as impurities in the sand's structure, influence the ultimate properties of the products. Clay structures, which have finer particle sizes than sand, can influence the relationship between the infill and cement and, consequently, the system's strength. Due to the layered silicate structure of clays, their propensity to incorporate water may cause a change in the cement-to-water ratio of the product, thereby affecting its workability. In the later phases of the hydration reaction, water losses or gains that may occur in the clay structure cause the product to expand and contract. This again leads to a decrease in the strength of the system. When all effects are taken into account, the clay content of the sand used in cement-based construction chemical products is of high importance for the final properties of the products [3, 7–9].

Silica is a fundamental constituent of several rock formations. Silica is found in several polymorphs, including α- and β-quartz, α- and β-cristobalite, coesite, and stishovite. At ambient circumstances, α-quartz is considered to be the most stable phase of silica. Quartz is a mineral that is widely distributed throughout the Earth's crust, constituting approximately 12% of its total weight [10].

Prior to its utilization in industrial applications, quartz need purification. A range of physical techniques are commonly employed for the purpose of eliminating diverse forms of impurities from quartz. The primary physical enrichment procedures that have been identified as significant are washing screening/classification, mechanical abrasion, gravimetric separation, as well as magnetic and electrostatic separation techniques. Flotation is a physicochemical process that involves the use of different collector and foamy reagents for the purpose of separation. Hydrometallurgical procedures are employed for the purpose of chemical purification [11,12]. The hydrometallurgical procedures encompass a tripartite sequence: firstly, the metal is extracted into a solution by leaching; secondly, the metal-enriched solution is separated from the solid waste or product; and finally, the required metal or compound is retrieved from the solution [13,14]. Biological approaches have garnered significant prominence in recent years, alongside chemical ones. The techniques known as bioleaching are well recognized for their cost-effectiveness and ecologically sustainable nature. These methods are particularly suitable for extracting metals from ores with low grades and for enhancing the enrichment processes of ores. Various approaches involve the utilization of heterotrophic bacteria in favorable habitats, as well as the application of certain organic acids such as oxalic, citric, ascorbic, and acetic acid, for the purpose of leaching [15-16].

Clay is not widely used as an ingredient in cement-based construction chemicals due to the performance and application issues it causes. The sand derived from the Marmara area contains many types of clays, including kaolin, illite, biotite, smectite, and palygorskite. These clays exhibit distinct mineralogical compositions and colors. By implementing an appropriate purification procedure, it is possible to deliver both clay and sand suitable for industrial consumption [17].

The depletion of natural resources, particularly in areas near human populations, restricts the availability of resources for the building industry. This, in turn, leads to rising costs that are passed on to consumers. The Marmara region's sand and clay reserves significantly impact several sectors, particularly the construction and ceramic industries, in terms of their quality, availability, and cost. The purification of raw materials that are acceptable for manufacturing is of greatest significance in relation to the sustainability impact, efficient utilization of natural resources, and the implementation of a circular economy. Accurately determining the usage requirements and characterizing purified raw materials is of utmost importance [18–20]. The primary locations of sand suppliers in the Marmara region, which is near Istanbul, include Arnavutköy, Çatalca, Çerkezköy, and Şile. The investigation involved the examination of sand samples gathered from different suppliers in the region. In the Arnavutköy region, the suppliers do not engage in any purification procedures for the sand. Conversely, the suppliers in Çatalça, Çerkezköy, and Şile undertake washing and drying procedures for the purification of the sand. The rapid growth in population and subsequent rise in housing demand have prompted the construction of residential zones throughout the Marmara Region. With the escalation of urbanization, numerous local sand quarries have become either inaccessible or restricted due to their proximity to residential areas. As a result, sand necessary for construction materials is increasingly sourced from far regions within the region or from outside the Marmara area, resulting in increased transportation costs and substantial carbon emissions. This issue highlights the imperative of assessing the usefulness and acceptable thresholds of current local sand reserves to guarantee economic efficiency and environmental sustainability. This study was undertaken to analytically evaluate regional sand resources and determine its appropriateness for the construction chemical uses. The study aimed to determine the clay concentration of sands suitable for usage in cement-based products within the construction chemicals industry. The clay content of the samples was determined using gravimetric technique, X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR) analyses within the framework of this study.

2. Experimental method

2.1. Materials and Methods

The sand samples included in this investigation were procured from several vendors located in Arnavutköy, Çatalca, Çerkezköy, and Şile. Sand samples with the same particle size distribution were used within the scope of the study. The gravimetric approach employed the ASTM E11 body Retsch brand 63-micron sieve and clean water acquired using the Pentair 40-400 device. The specimens were subjected to a drying process using a Binder brand oven. Prior to examination, the samples designated for X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) were subjected to grinding using a Retsch MM400 grinder in order to decrease the grain size. The X-ray diffraction (XRD) technique was employed to describe the crystalline phases. A Philips PANalytical X'Pert-Pro X-ray diffractometer was utilized, operating at 40 mA and 30 kV, with a step size of 0.02. The scanning range for all samples was set at $2\lambda = 5–70$. The samples were subjected to Fourier-transform infrared spectroscopy (FTIR) measurements using a Perkin Elmer Spectrum 100 (United States) series equipment equipped with attenuated total reflection (ATR) mode.

2.2. Gravimetric Method

The objective of the gravimetric approach is to effectively separate the quartz (SiO2) and clay components present in sand samples, estimate the quantity of clay, and subsequently isolate and analyze the clay in accordance with its characterization.

The gravimetric approach involves the measurement of a certain quantity of the sample, which is subsequently combined with a predetermined volume of water. The substance is sieved using a 63-micron mesh to ensure the removal of any lumps, with the use of a spatula to facilitate the process. Following the completion of this procedure, whereby the process is continued until the water achieves clarity, the sand is subsequently transferred onto a watch glass that has been tared, and subsequently subjected to a drying process. The aqueous solution containing clay particles is subjected to a drying process by passing it through a filter paper with a predetermined weight. Consequently, masses of sand and clay are measured distinctly and an estimation of the clay content is subsequently derived. The phases that have been separated can be later used for characterization purposes [21,22]. The formula employed in the determination of clay and quartz phases by the gravimetric technique, as previously elucidated, is as follows:

$$mclay = m1 - m0 \quad (1)$$

$$mquarts = m3 - m2 \quad (2)$$

where;

m0 represents the mass of the filter paper,
m1 represents the combined mass of the filter paper and clay after being subjected to heat,
m2 represents the mass of the glass slide
m3 represents the mass of the dried quartz contained within the glass slide

2.3. XRD and FT-IR Analysis

The characterization of clays by XRD can be challenging when they consist of a combination of calcite or quartz, mostly due to their layered structural composition. While XRD analysis is adequate for identifying clays based on their distinctive peaks within the 2θ 0-10-degree range, relying solely on the Rietveld technique is insufficient for quantitative analysis [23–25]. The materials analyzed in this study were subjected to a combined analysis using XRD and FTIR techniques. In the gravimetric approach, clay may be immediately evaluated after separation from the quartz phase. However, for other materials, grinding is necessary prior to analysis.

3. Results

The simulation samples were prepared with the addition of external clay to sand samples that contained quartz with a purity level over 99%. Subsequently, the gravimetric technique was employed to analyze these samples. The computation yielded a high level of accuracy in samples characterized by a high clay percentage; however, discrepancies were seen in cases when the clay content was exceptionally low. Both approaches are suitable for the characterization of the clay that is extracted from the body. Images showing the experimental flow are given in Figure 1.

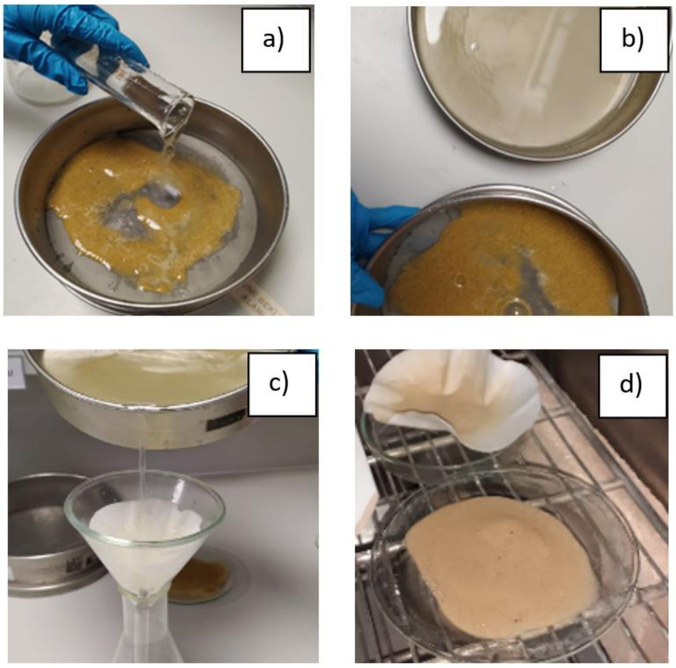


Fig. 1. Gravimetric method shows the separation of clay and quartz (a) adding water to sand; (b) separation of quartz and clayish water; c) separation of clay and water with filter paper; d) drying of separated quartz and clay in the oven.

XRD diffractogram of the samples, both with and without clay, as provided by the supplier without any purification process, is depicted in Figure 2. The angle of diffraction, 2θ, is used to represent the scattering angle. The most prominent peaks associated with quartz are detected at 20.8 and 26.5 degrees, while less prominent peaks are recorded at 36.5, 39.4, 40.3, 42.4, 45.7, 50.1, 54.9, 60.01, 64.1, and 68 degrees. The clay-free sample was subjected to the Rietveld examination, revealing that it included 97% quartz and 3% impurities in the form of trace zeolite, coesite, and lazurite integrated within its structure [26,27]. Differences between the diffractograms of samples with and without clay appeared depending on the clay and feldspar content. The clay mineral known as montmorillonite, which has a diffraction peak at a 2θ angle of 5.8°, belongs to the smectite group [28,29]. Biotite, which has a diffraction peak at a 2θ angle of 8.8°, is classified as a member of the mica group and is considered a clay mineral [30]. Both constituents are referred to as phyllosilicates and exhibit a stratified arrangement. The residual constituents observed in the diffractogram comprise several types of feldspars, including albite, microcline, anorthite, and anorthoclase. The area between 2θ 27°-29° has the most prominent peaks associated with feldspars.

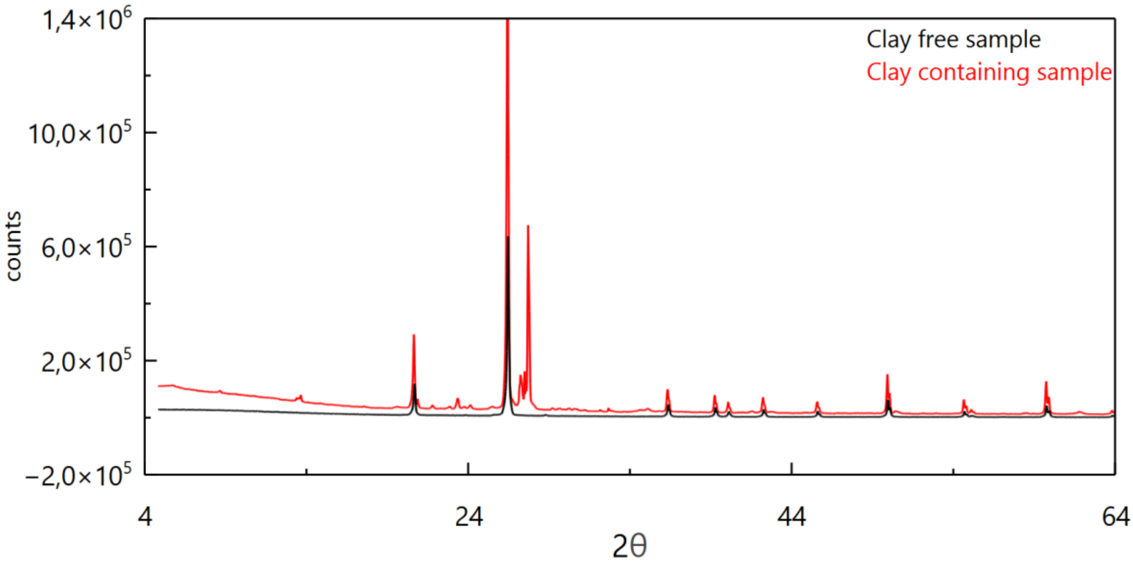


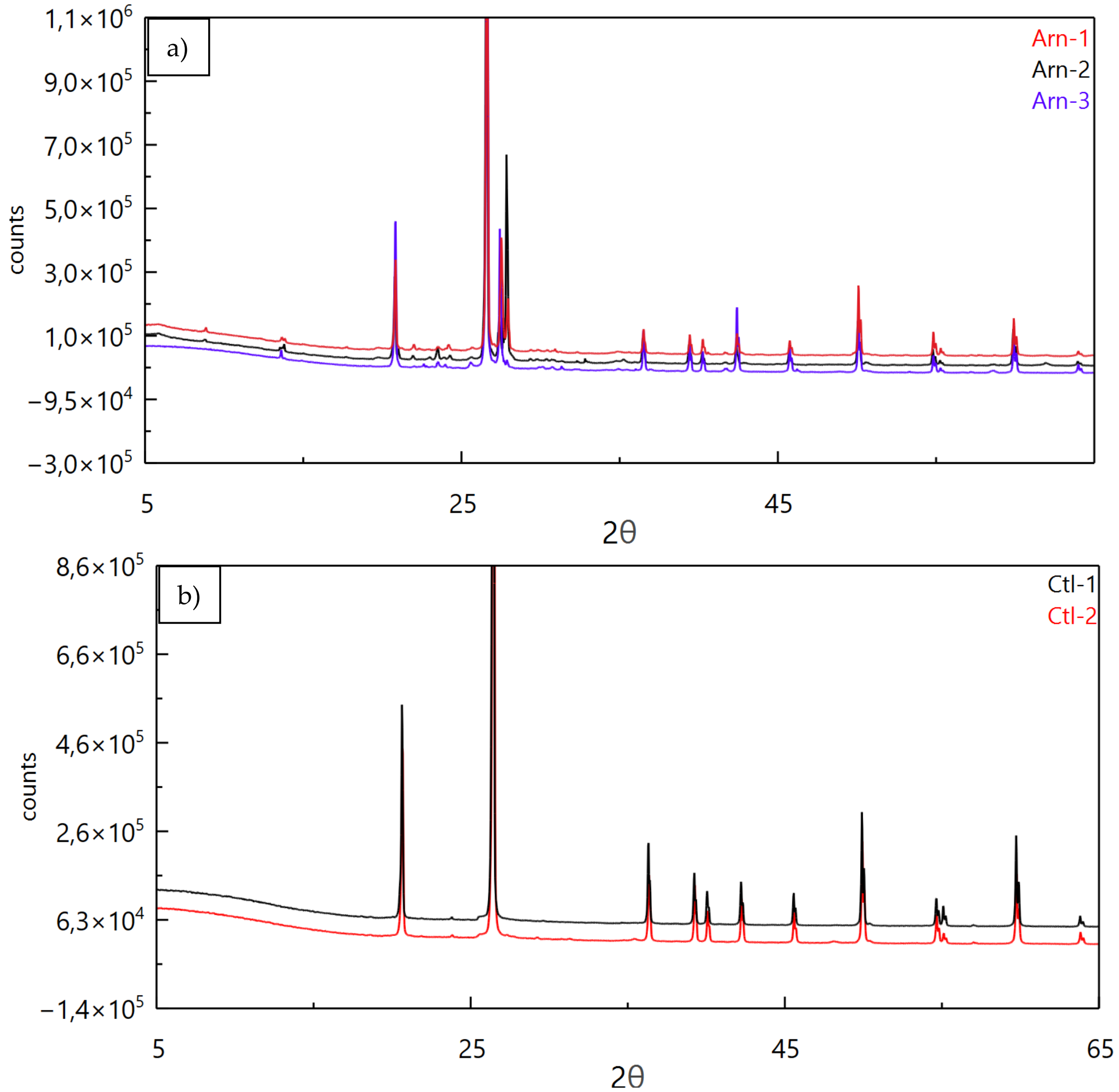
Fig. 2. XRD diffractograms of clay containing and clay-free samples.

XRD analyses were conducted on all samples obtained from sand suppliers located in the Marmara region, specifically Arnavutköy, Çatalca, Çerkezköy, and Şile. The resulting diffractograms are shown in Figure 3. The nomenclature for the sample is presented in Table 1. Samples were procured from several suppliers or different sources within the same supplier, all originating from the same procurement area. Each sample was assigned an identification number.

Table 1. Sample nomenclature.

Location	**Sample Name**
Arnavutköy	Arn
Çatalca	Ctl
Çerkezköy	Crk
Şile	Sl

Upon examination of the Arn samples, it is evident that Arn-3 exhibits the lowest levels of impurities in relation to clay and feldspar composition. Subsequently, Arn-2 and Arn-1 display progressively higher amounts of impurities. The Ctl samples have a significantly greater proportion of quartz in comparison to the Arn samples. The Crk-1 sample has a greater concentration of clay and feldspar compared to the Crk-2 sample. Conversely, the Sl samples have a significantly larger proportion of quartz in comparison to the Crk samples. XRD diffractograms were analyzed using the Rietveld method. Given that the samples consisted of distinct clay compositions, a comparison was made by evaluating their quartz content, which subsequently yielded confirmed outcomes.



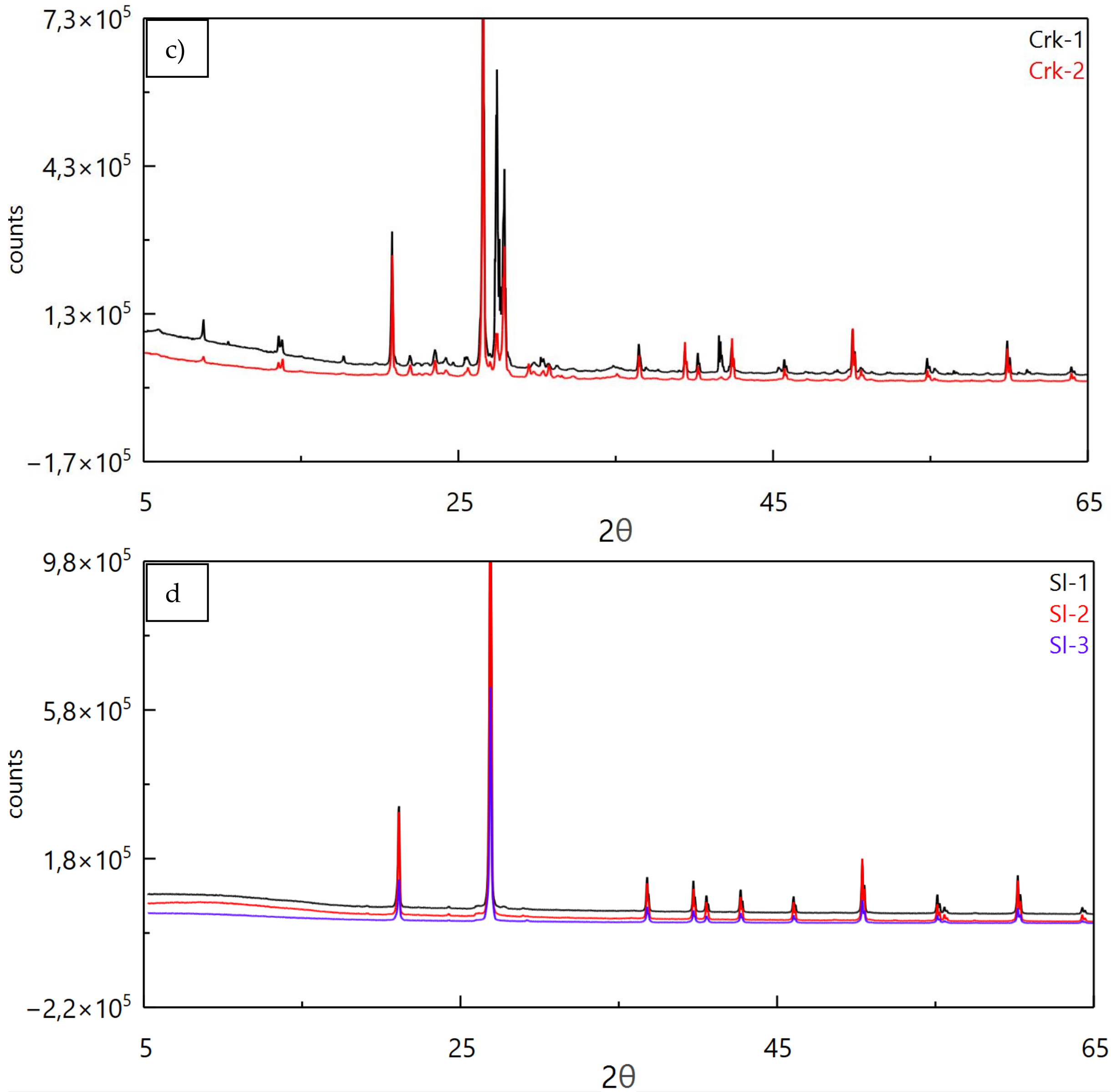
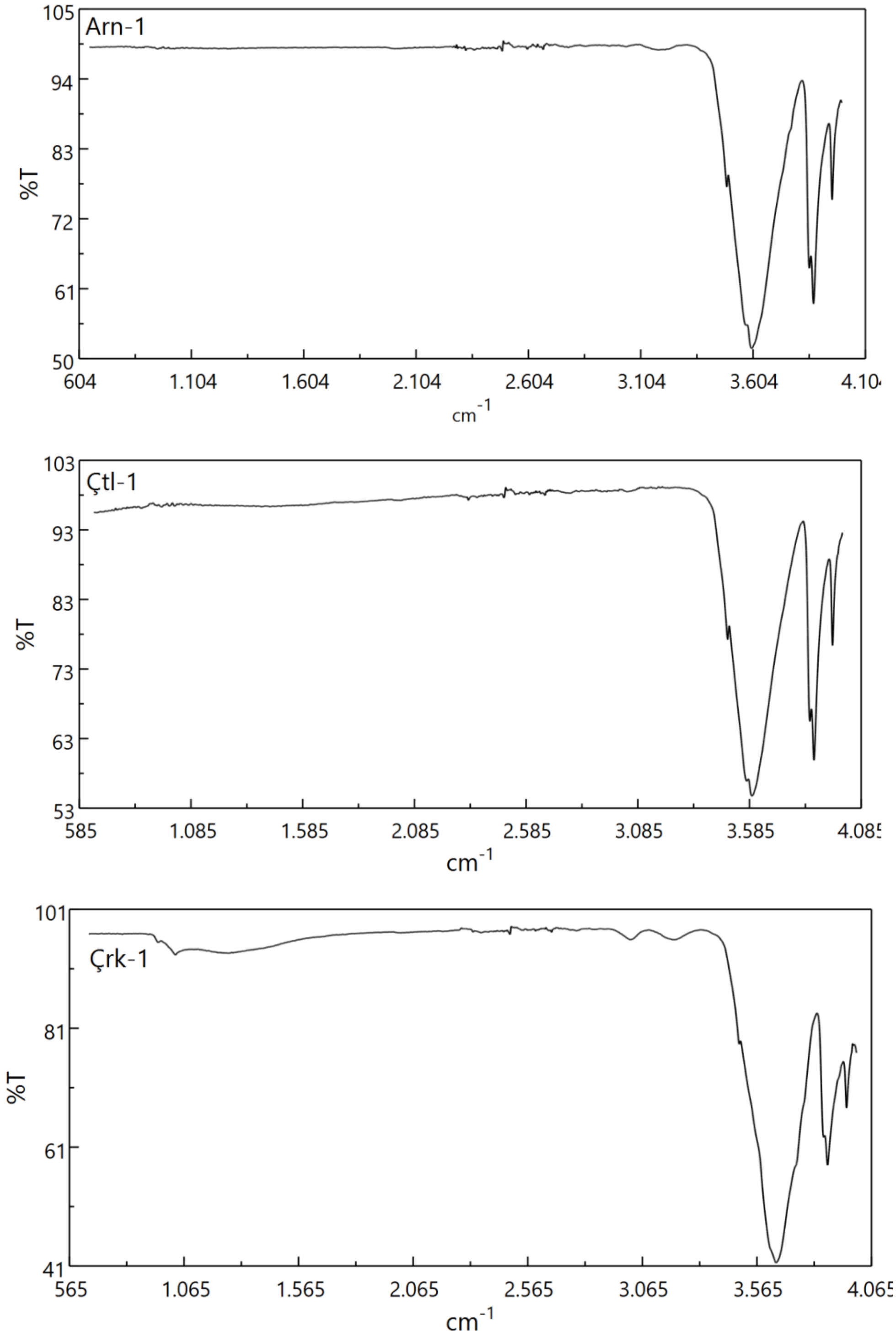


Fig. 3. XRD diffractograms of samples obtained from (a)Arnavutköy; (b)Çatalca; (c) Çerkezköy; (d) Şile.

The XRD diffractograms of the Arn samples in Figure 3-a exhibit variations in clay and feldspar compositions among the three samples. The clay contents are ranked in the following order: 1, 2, and 3. The intensity of the peak associated with mica group clay varies, particularly in the 2θ 8° region. Feldspar peaks were detected at the temperature range of 27 to 29°. Upon examination of the diffractograms of the Ctl samples in Figure 3-b, it was seen that there was no discernible peak development in the clay areas at 5 and 8°s. Furthermore, the absence of any peak development within the range of 27-29° indicates the presence of feldspar. Figure 3-c displays the XRD diffractograms of the Crk samples. The Crk samples had the greatest clay and feldspar concentration among all the samples taken from different areas. Peak formation was detected in all clay and feldspar regions, with peak intensities above those of other samples.mThe XRD diffractograms of Sl samples in Figure 3-d exhibit a comparable profile to that of Ctl samples, indicating a low clay concentration.

The presence of clay peaks in XRD analyses was verified using FTIR analysis, which revealed wide peaks attributed to the low concentration of clay in the structure and its largely amorphous nature. Figure 4 presents the FTIR spectrum of the sand samples labeled as number 1, which were collected from various regions.



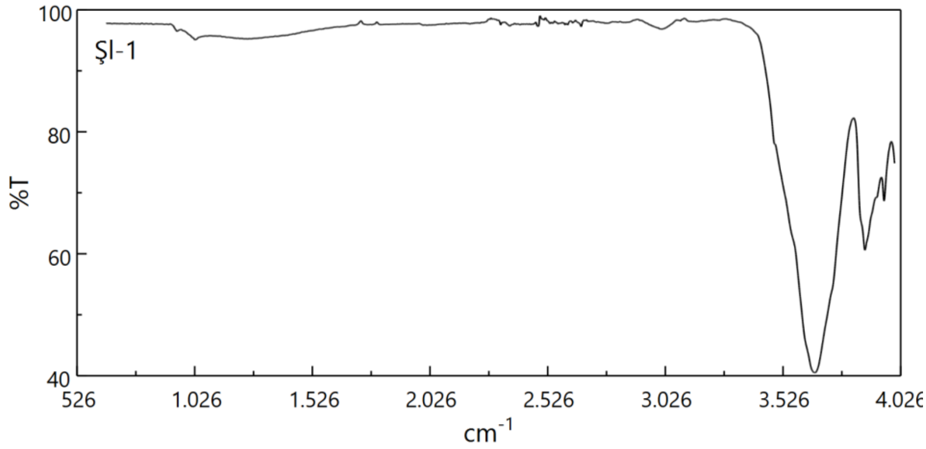


Fig. 4. FTIR spectrum of samples (Arn-1; Ctl-2; Crk-1; Sl-1)

Peaks at the wave number of 3600 cm^{-1} can be observed, dependent upon the clay concentration. Furthermore, it was noted that the prominent peak attributed to the Si-O-Si band at a wave number of 1000 cm^{-1} was predominantly caused by quartz. However, this peak exhibited a rightward shift when the clay content exhibited an increase. The observed shift, which appears at a wave number of 1000 cm^{-1}, becomes stronger when the spectrum is normalized with respect to 1000 cm^{-1}, hence accentuating the alteration at 3600 cm^{-1}. Figure 5 presents the FTIR spectra that have been normalized to a value of 1000 cm^{-1}.

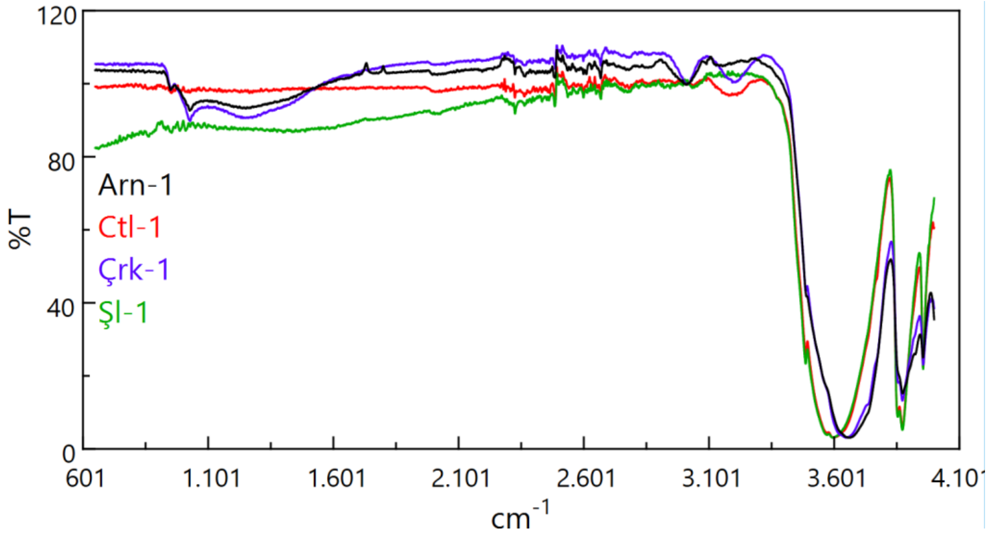


Fig. 5. FTIR spectrum of samples normalized to 1000 cm^{-1}

4. Conclusion

The research effort collected samples from several sand suppliers located in the Marmara region, specifically targeting the areas of Arnavutköy, Çatalca, Çerkezköy, and Şile. Except for the samples acquired from Arnavutköy, all samples underwent a purification procedure which encompassed washing and drying stages in sand facilities. In this work, the gravimetric technique, FTIR analysis, and XRD were employed to characterize sands containing clay content that does not significantly impact rheology, with the intention of utilizing them in the production of construction chemicals. The study revealed that the gravimetric approach is a viable option for situations characterized by insufficient laboratory facilities and the need for clay-quartz separation. Furthermore, it has been found that the utilization of XRD analysis, in conjunction with the Rietveld method, yielded highly accurate outcomes in the quantification of quartz and feldspar constituents. The utilization of FTIR analysis is deemed helpful in cases when a substantial dataset has been previously examined and a well-established methodology for determining clay content exists. In the context of conducting technique studies for FTIR analysis, it is advisable to generate simulation samples by blending high purity quartz with a clay of known composition in certain ratios. Subsequently, the spectrum of the given sample may be compared to the spectra of the simulation samples.

Furthermore, despite the absence of a purification step, Arn samples exhibit reduced quantities of clay and feldspar compared to processed Crk samples. The Ctl and Sl samples have a greater concentration of quartz compared to all other samples, and their impurity levels are exceptionally low. Although the Arn samples were not purified as part of the investigation, it was found that the clay concentration of the samples fell within the acceptable range for the production of cement-based construction chemicals. Materials derived from natural sources often exhibit a range of contaminants. Adherence to quality control requirements is crucial when utilizing natural resources in the manufacture of industrial materials. After evaluating the factors of sustainability and proximity in production, it was determined that the Arnavutköy area in Istanbul is suitable for sourcing sand material with limited clay content. The absence of purification needs, such as the need for washing and drying, might result in a cost benefit. When utilizing this information in the future, it is advisable to consider the present reserve status, mineral processing licenses, and transportation expenses.

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