Araştırma Makalesi / Research Article

Investigation of Hydrophobicity in Calcium Stearate-Epoxy Based Composite Materials

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ABSTRACT: Hydrophobic surfaces are essential for enhancing the longevity and performance of products across various sectors, particularly in construction, automotive, electronics, and medicine. This characteristic minimizes maintenance expenses by establishing a natural barrier against pollution and corrosion, hence prolonging the lifespan of the materials. Moreover, implementing this technology provides novel methods for attaining energy efficiency and environmental sustainability objectives. This research introduced a composite material formulated with calcium stearate (CS) and epoxy, designed as a coating solution to enhance hydrophobic qualities on surfaces. This technique is intended to create a formulation that integrates the water-repellent characteristics of CS with the structural resilience of epoxy for application on various surfaces. The contact angle of the composite, derived from adding CS into epoxy at concentrations of 1%, 2%, 4%, 6%, 8%, and 10% by weight, was assessed against pure water. Neat epoxy has a hydrophilic structure with a contact angle of 70.5°, whereas the composite containing 8% CS demonstrates the highest hydrophobicity, with a contact angle of 117.5°. Furthermore, adding CS led to a decrease in Shore D hardness values.

Keywords: Composite, Calcium stearate, Hydrophobic, Hydrophilic, Contact angle

1. INTRODUCTION

Today, due to their water resistance, corrosion resistance, and antifouling properties, hydrophobic coatings have become necessary and have a wide range of uses. Calcium stearate (CS) has found a place in many studies in this context due to its hydrophobic properties. CS is a water-insoluble metallic stearate salt formed by the combination of stearic acid and calcium ions. It is frequently used in industry as anti-wear, plastic stabilizer, water-repellent additive and surface coating additive (Wang et al., 2020; Li & Yao, 2011). In a study where it was used as a PVC additive due to its high thermal stability, it was stated that CS delayed thermal degradation and preserved the structural integrity of the polymers (Wang et al., 2020). In addition, it has been observed that when used in cement and concrete-based building materials, CS reduces the water absorption rate by reducing the number of pores in the structure and thus increases the resistance to external factors in the long term (Maryoto, 2017; Chen et al., 2022; Lv et al., 2022). However, its use in high-performance polymer matrices like epoxy, especially at the micro-scale level, remains limited and underexplored.

Due to its low surface energy, CS exhibits hydrophobic properties. When used as a coating, it increases the contact angle against water and, therefore, the water resistance. In a study, nano-sized CS-added coatings were prepared via a single-step dipping method, and contact angles of over 150° against water were obtained (Bai et al., 2023). In the same study, it was revealed that the mentioned coatings maintained their hydrophobicity for a long time (Bai et al., 2023). Superhydrophobic coatings containing CS have high resistance to thermal shock, organic solvents, and UV rays (Bai et al., 2023). It has been observed that the coated surfaces retain their hydrophobic properties even after 100 times of sandpaper abrasion and 300 times of tape peeling test. Therefore, it has been suggested that CS can be used in the production of low-cost and environmentally friendly water-repellent coatings (Bai et al., 2023). However, most literature focuses on nano-structured additives or chemical surface modifications to achieve hydrophobicity in epoxy systems. For example, Chen et al. (2024) showed that the contact angle increased from 60.43° to 114.66° by adding 0.89 wt% GO, significantly improving the corrosion resistance.

Ari (2025) evaluated epoxy composites filled with ignimbrite and pine waste and found that the contact angle increased up to 113.79°, while mechanical properties decreased with increasing filler content. These findings highlight the need for studies that optimize both hydrophobicity and mechanical integrity, particularly with industrially relevant micro-fillers like CS.

CS is applied with different methods to provide a hydrophobic surface for different materials. When added to concrete mixtures, it increases the material's service life by reducing the water absorption rate and chloride ion penetration (Chen et al., 2022). In the study, it was shown that the optimum ratio of CS used as an additive at different rates was 4% and that when used at this rate, it significantly reduced the water absorption rate (Chen et al., 2022). It has been suggested that when 3% CS is added to ultra-high molecular weight polyethylene (UHMWPE), the dry wear resistance of UHMWPE increases by 4 times, the friction coefficient decreases by 1.2 times, and therefore it can be used as a solid lubricant (Panin et al., 2015).

Epoxy resins are thermoset polymers used as matrix materials in protective coatings, structural adhesives, and composite materials due to their high mechanical strength, chemical resistance, and high adhesion properties (Xavier, J.R., 2020). Epoxy coatings are frequently used to protect metallic material surfaces from corrosion. Epoxy coatings modified with various metal oxide nanopowders greatly reduce water permeability and corrosion (Xavier et al., 2024). In addition, thin and porous coatings may increase the contact of the substrate material with the water environment and negatively

affect long-term durability. Therefore, different methods may need to be applied to increase the hydrophobic properties of epoxies (Xavier et al., 2024). In one study, fluorine-based additives, nanoparticles that increase surface roughness, and silicon-containing modifications were used to increase the water repellency of epoxy-based coatings (Chang et al., 2022). In another study, as a result of modification of ZIF-8 particles with DMBIM, the contact angle increased to 124.9°, and the hydrophobicity of the coating and thus the corrosion resistance in acidic, basic, and salty environments was significantly increased (Chen et al., 2025). In a study on epoxy composite coatings produced with clay modification, it was observed that water permeability was reduced, and therefore, the coatings maintained their long-term stability (Verma et al., 2024). Aparna et al. (2022) provide a comprehensive review of superhydrophobic epoxy nanocomposites, detailing how fabrication methods, surface roughness, and low-energy additives such as silica and carbon-based nanostructures contribute to water repellency. However, these methods often require complex chemistry and high costs. Doganci and Sevinc (2023) showed that epoxy coatings reinforced with stearic-acid-modified alumina nanoparticles reached contact angles over 150° and demonstrated superior corrosion resistance and surface roughness, especially on galvanized steel. This validates the relevance of roughness-enhancing micro-fillers for practical applications.

This study aimed to investigate the effects of different amounts of CS addition to increase the hydrophobic characteristics of epoxy surfaces. In the literature, it has been demonstrated that CS has water-repellent properties in concrete and cement structures, but its effect on epoxy surfaces has not been investigated sufficiently. Improving the hydrophobic properties of epoxy coatings can increase corrosion resistance and extend the service life of coatings by reducing water absorption. In this context, epoxy composites with CS additive were produced and their contact angle against water and hardness were analyzed. The results of this study will contribute to the development of low-cost, environmentally friendly, and high-performance hydrophobic surfaces. By evaluating CS content ranging from 1 to 10 wt%, this study provides one of the first systematic efforts to quantify the relationship between CS loading, hydrophobic response, and Shore D hardness in epoxy composites, thus offering practical guidelines for material design and application.

2. MATERIALS AND METHODS

2.1 Materials

Epoxy is used as the matrix material and reinforced with CS to increase its hydrophobic properties. MGS LR135/LH135 lamination epoxy set was provided by Dost Kimya Company/Türkiye. According to the information received from the supplier company, the mixing ratio of the resin LR135 and hardener LH135 is: 100:35 ±2 g, and has a working time of 30-45 minutes, depending on the ambient temperature. Its density is 1.1-1.23 g/cm^3, and its viscosity is 1000-1500 mPas.

Melos Inc./Türkiye provided the CS 3701 particles. The company claims that CS3701 is obtained from the reaction of stearic acid and lime: $2C_{17}H_{35}COOH + CaO \rightarrow (C_{17}H_{35}COO) 2Ca + H_2O$. The appearance of the powders is white powder, and the melting point is 150,0 ±2,0 °C.

2.2 Particle Size Distribution Measurements

Particle-size distributions of CS particles were determined using the dynamic light scattering (DLS) method by a Malvern Mastersizer 2000 instrument, shown in Fig. 1 (Malvern Panalytical Co., UK), equipped with a dry measuring apparatus. First, the vacuum and compressed air flow were started to clean the measuring system for about 2 minutes. Subsequently, an automatic background

reset was executed while the airflow persisted. The dry sample was thereafter filled in the sample section, and the sample feeding rate was augmented until the laser obscuration level attained 4-5%. Subsequently, automatic measurements commenced, and the average values were taken by conducting each analysis thrice.

Figure 1. Malvern Mastersizer 2000 particle size analyzer instrument

2.3 Sample Preparation

Flat surface composite samples were prepared to measure contact angle against water and Shore D hardness. Composite materials were produced with different CS additive ratios of 0%, 1%, 2%, 4%, 6%, 8%, and 10% by weight. First, the epoxy sample without additive was poured into the mold after mixing the resin and hardener components in a 100:35 ratio by weight with a mechanical mixer for 5 minutes. To prepare the CS-added samples, firstly, the powder CS particles in the determined ratios were mixed with the resin component for 10 minutes. Then the resin-powder mixture was mixed with the hardener for 5 minutes.

Composite samples were produced by pouring the epoxy mixtures into circular polyethylene molds with a diameter of 25 mm. To prevent any effects from the mold surface texture on subsequent measurements, the analyses were performed on the top surface of each composite, which was not in direct contact with the mold material. The curing was performed in two steps, based on manufacturer recommendations: initial curing at room temperature (approximately 23 ± 2 °C) for 24 hours, followed by post-curing at 80 °C for 30 minutes. During room temperature curing, the samples were isolated from environmental pollutants by placing them inside a closed desiccator. After curing processes, the sample surfaces were ground sequentially using sandpapers with grit sizes of 800, 1000, 1200, and 2000 and then polished with the 1 µm alumina solution. Before conducting measurements, samples were thoroughly cleaned using aqueous detergent, followed by rinsing with distilled water in an ultrasonic cleaner.

2.4 Surface Roughness (Ra) Measurements

The surface roughness (Ra) of the top surfaces has been assessed, where contact angles were measured on free surfaces not in touch with the mold, as surface roughness significantly influences the contact angle. The measurements were conducted using the Nanovea ST400 3D optical surface profilometer (AnMo Electronics Corporation, New Taipei City, Taiwan). The profilometer is shown in Fig. 2. The profilometer scanned the surface at a velocity of 5 mm/s throughout a 1 mm x 1 mm region, and the measurement data were evaluated via MountainsMap software. The mean of five measurements for each sample was recorded.
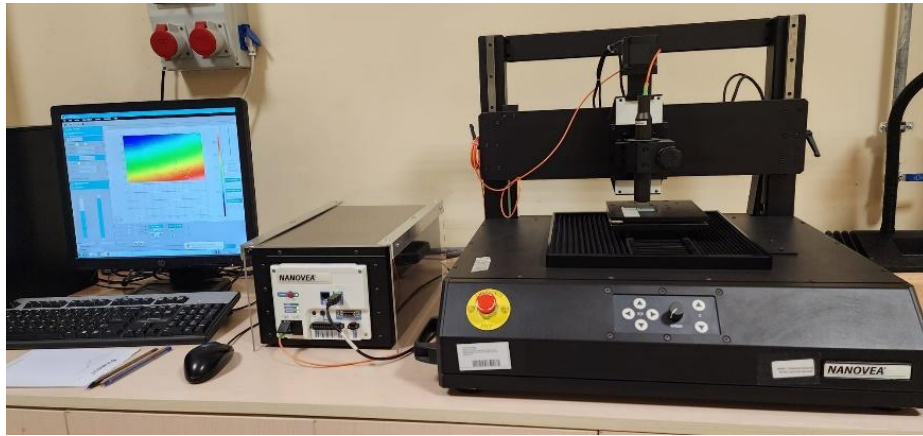
Figure 2. The 3D Optical profilometer device is used in surface roughness (Ra) measurements

2.5 Contact Angle Measurements

The wettability (or conversely, hydrophobicity) of the samples was determined by contact-angle (θ) measurements using the sessile drop method at room temperature by a One Attension theta optical goniometer (Biolin Scientific Co., Espoo, Finland). The device is shown in Fig. 3. Before initiating θ measurements, the surfaces of the samples were cleaned by washing with aqueous detergent. Then, it was rinsed with distilled water. Finally, the cleaned samples were left to dry in a vacuum oven at 60°C for 24 hours. Subsequently, the θ measurement was started by settling about 5 μL of distilled water onto the sample using a Hamilton injector. The value at the 2nd second after the water drop was released onto the sample surface was recorded. The θ measurements were taken from three different points on the surface of each sample, and the values closest to the average were recorded.
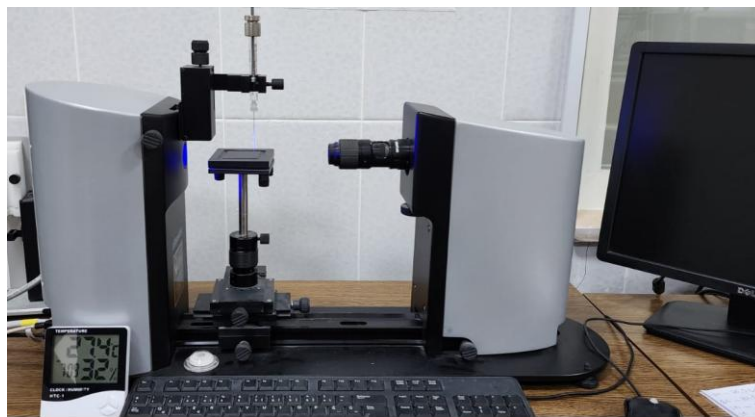

Figure 3. One Attension theta optical goniometer used for water drop contact angle measurement

2.6 Hardness Tests

5 hardness values were measured from the surface of each sample, and the average was recorded. In this study, Shore D hardness measurements were conducted using a Shore D durometer as per ASTM D2240, which is also commonly employed in polymeric and composite materials research due to its reliability, ease of use, and wide industrial acceptance (Dinesh et al., 2020; Ganapathy et al., 2021; James et al., 2020). Barcol hardness testing, while also could be preferred, is more commonly applied to composite materials with fiber reinforcement (Ceritbinmez et al., 2021; Ceritbinmez et al., 2022).

3. RESULTS AND DISCUSSION

3.1 Particle Size Analysis

The d_{10}, d_{50}, and d_{90} values (Fig. 3) show particle sizes, and 10%, 50%, and 90% of the sample amount are smaller than these particle sizes, respectively. For example, d_{90} of the CS sample means that 90% of the sample consists of particles with a size under 16.8 μm.
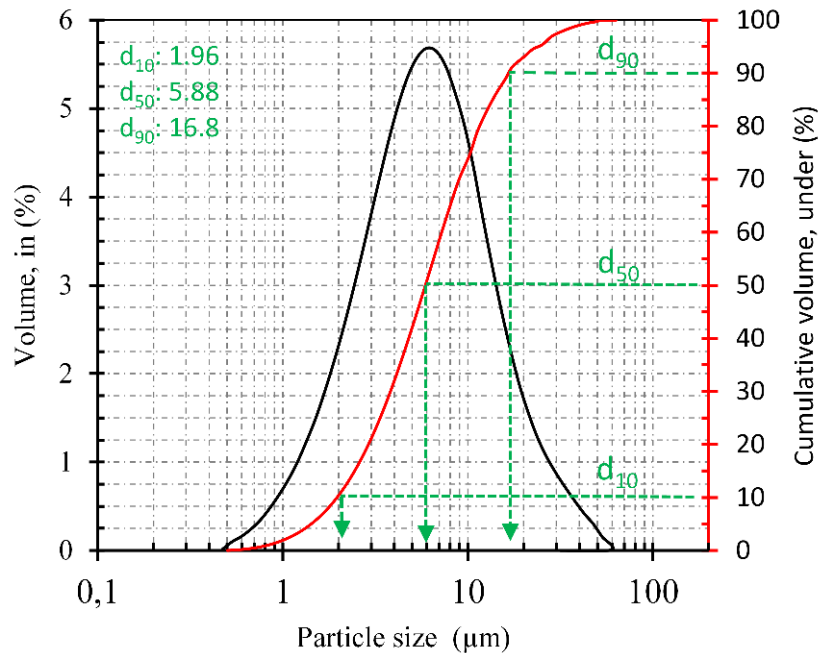

Figure 4. Particle size distribution analysis of CS particles

Figure 4 revealed that the CS powder sample consisted of very fine-sized particles. Namely, the particle size distribution varied uniformly between 0.5-60 μm, and the d_{90} value of the sample was determined as 16.8 μm. According to Figure 4, the particles constituting the CS powder were mostly concentrated around the 6 μm size. Thus, the average particle size of the sample can be expressed as approximately 6 μm. The d_{50} value of the sample, being 5.88 μm, is also an indication of this. In addition, the CS powder sample has a narrow size distribution at the medium scale, as can be seen from the low slope of the cumulative undersize curve. As a result, according to the particle size distribution data, it can be said that the CS sample can form topographically homogeneous dispersions with epoxy-like resins under appropriate conditions.

3.2 Hydrophobicity of Composites

The hydrophobic characteristics of epoxy were assessed by measuring the contact angles of the specimens produced with water, utilizing various ratios of CS additives (0%, 1%, 2%, 4%, 6%, 8%, and 10%). Due to its effect on contact angles, the surface roughness of the composites was measured and is shown in Table 1. Fig. 5 depicts the effect of different CS additions on contact angles. The contact angle of pure epoxy with water is shown in Fig. 5(b), and the angle of 70.5° indicates that it has a hydrophilic structure. Generally, surfaces with a contact angle below 90° are termed hydrophilic, while those above 90° are termed hydrophobic (Ma et al., 2007). Due to CS's low surface energy, it exhibits hydrophobic properties (Bai et al., 2023), and the contact angles have significantly increased with the addition of CS (Fig. 5). With a 1% CS addition, the contact angle increased to 104.7°, followed by small fluctuations in contact angles at 2%, 4%, and 6% (101.5°, 102.0°, and

107.9°, respectively). The maximum contact angle attained was 117.5° using 8% CS. Despite anticipating an increased contact angle with the addition of 10% CS, the contact angle marginally fell to 112.5°, likely due to agglomeration effects. The reduction in contact angle with a high CS content ratio may be related to pore formation resulting from the epoxy's inadequate wetting of CS particles with low surface energy in specific regions.
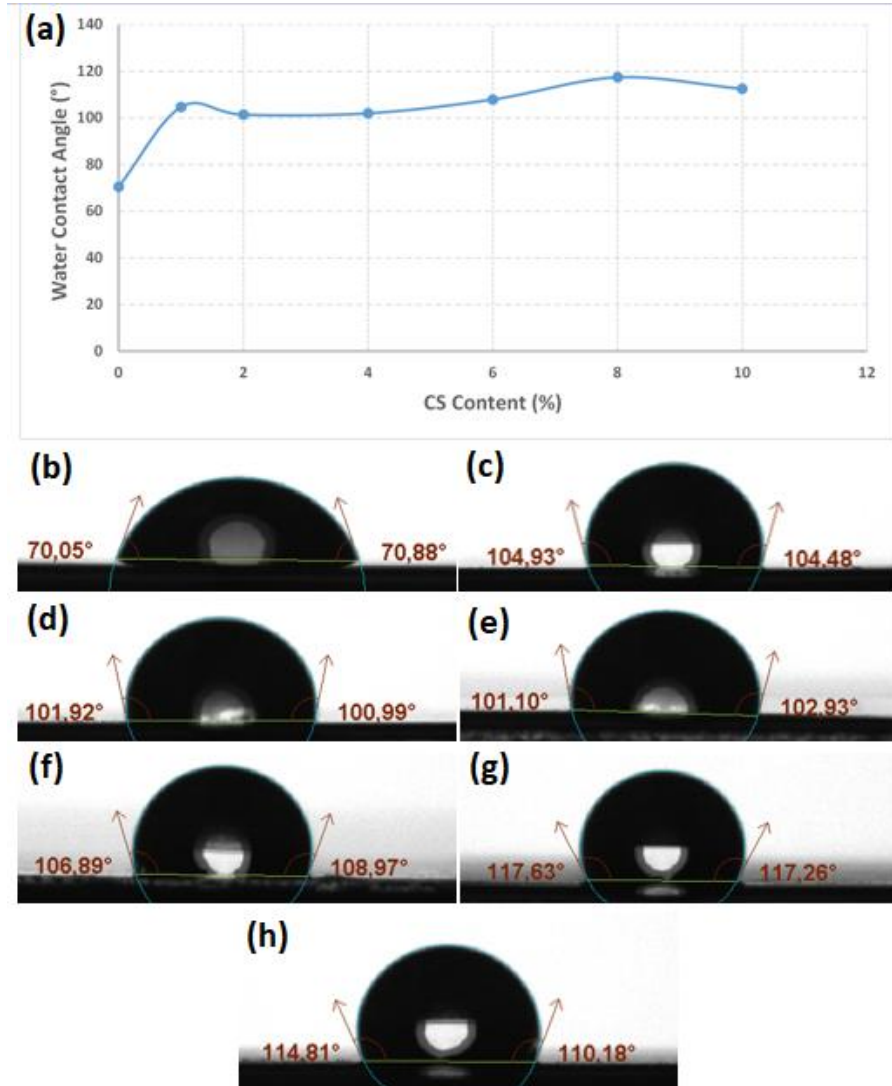

Figure 5. Contact angle measurement results against water (a) the graph illustrating the variation of contact angles with water with different CS ratios, and contact angles of specimens with (b) 0%, (c) 1%, (d) 2%, (e) 4%, (f) 6%, (g) 8% and (h) 10% CS additions

A study on coatings using CS nanoparticles found contact angles of 150° against water, indicating good mechanical durability and resistance to UV radiation, thermal shock, and organic solvents (Bai et al., 2023). Similarly, adding CS to cement and concrete lowers the amount of water that can penetrate structures and the amount of chloride ions that can get in (Chen et al., 2022; Lv et al., 2024).

It has been demonstrated in various studies that the hydrophobic properties of epoxy can be enhanced using different methods. It has been reported that the contact angle of epoxy surfaces modified with ZIF-8 and DMBIM increased to approximately 124.9°, and the corrosion resistance in harsh environments such as acid, alkali, and saline water was significantly improved (Chen et al.,

2025). Additionally, the use of nanoparticles such as clay-modified silico-graphitic carbon (Verma et al., 2024), graphitic carbon nitride, and manganese dioxide (Xavier et al., 2024) has improved the hydrophobicity and mechanical properties of epoxy surfaces.

3.3 Hardness of Composites

In order to observe the effect of CS additives on the mechanical properties of epoxy, Shore D hardness values of the samples were examined. While the hardness of neat epoxy is 89.5, the hardness of the composite with 1% CS additive is 89.6, and these two values are quite close. However, as can be seen in Fig. 6, the Shore D hardness values of the composites with 2% or more CS additives are visibly lower. Especially after adding 4% CS, the hardness value (approximately 70.9) significantly fell below the hardness value of pure epoxy. At the 6% and 8% ratios, the hardness decreased to approximately 61.3 and 63.7, respectively, while at the 10% ratio, it slightly increased again to approximately 69.4.

The general mechanical properties and hardness of epoxy resins are directly dependent on the type, amount, and distribution of fillers added to the polymer matrix (Pham & Marks, 2002). It is stated that if the filler materials show a homogeneous distribution within the polymer matrix, the mechanical properties will improve. However, the addition of high amounts of filler materials acts as a defect in the structure and negatively affects the mechanical properties (Zhou et al., 2008; Panin et al., 2015). CS is a material with low surface energy and may not have been wetted enough in some areas when mixed with epoxy. In this case, it is possible that the dry areas were trapped in the structure during the curing of the epoxy and caused the hardness to decrease by creating voids after full curing.
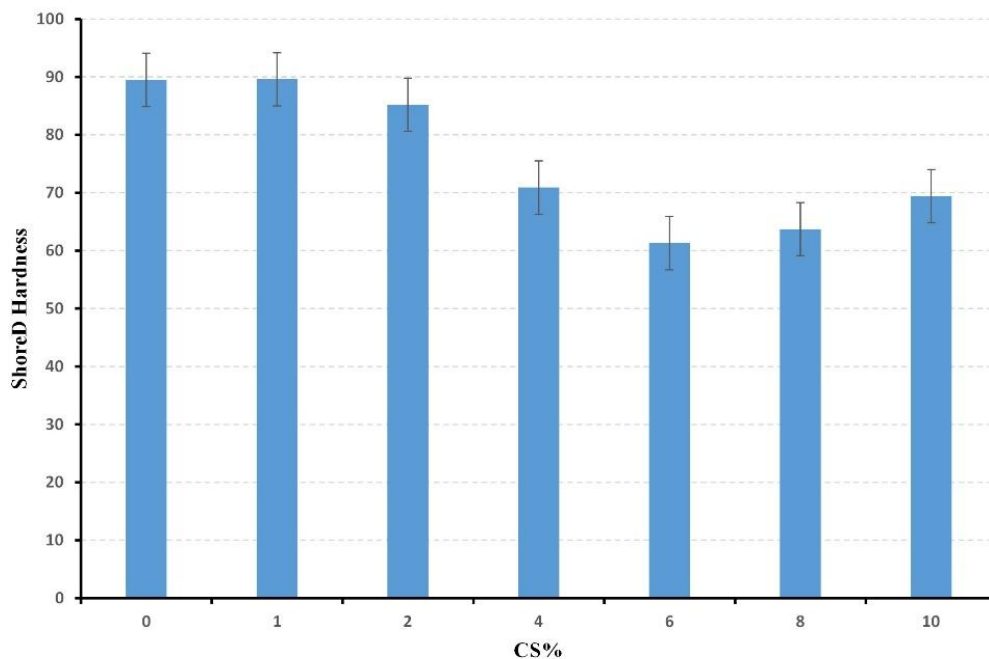

Figure 6. Shore D hardness values of neat epoxy and Epoxy-CS composites

Panin et al. (2015) state that the hardness values decrease with the high addition of CS to the UHMWPE matrix, and this situation is due to the soft structure of CS (Panin et al., 2015). The reduction in hardness with increased CS concentration aligns with findings reported by Zhou et al. (2008), who studied carbon nanotube-epoxy composites. They observed that particle agglomeration at higher filler ratios resulted in stress concentration points within the epoxy matrix, which facilitated

early crack initiation and subsequent mechanical failure. This perspective is additionally corroborated by prior research that highlights the essential function of filler dispersion in mechanical reinforcement. Onuegbu and Igwe (2011) examined polypropylene composites filled with snail shell powder and found that larger filler particle sizes, along with increased agglomeration, led to diminished hardness values due to decreased interfacial adhesion and stress transfer. Dinesh et al. (2020) similarly reported that jute fiber-epoxy composites reinforced with coarse rosewood dust demonstrated lower Shore D hardness than those with fine Padauk wood dust, a phenomenon attributed to inadequate dispersion and the creation of filler agglomerates that functioned as weak points within the matrix. These data validate the correlation identified in our study between filler agglomeration and diminished hardness, highlighting the necessity of attaining a uniform filler distribution to preserve the integrity of the composite structure.

Similarly, our results suggest that at elevated CS concentrations, agglomerated particles and associated void formation likely created structural heterogeneities within the epoxy matrix, thus leading to a noticeable decrease in hardness. The decreasing trend of hardness values in our study confirms this literature information. It is thought that the homogeneous distribution of particles is optimal in the sample with 1% addition, where the hardness is highest. However, with the increase in the CS ratio, the agglomeration of particles increases, leading to the formation of non-homogeneous regions within the matrix and a decrease in hardness. The slight increase in hardness at the 10% ratio may be the result of different structural changes caused by the higher filler content, but it is still quite low compared to the hardness of the sample containing 1% CS.

Table 1. Surface Roughness (Ra), Shore D Hardness, and Contact Angle Values of the Composite Samples

Sample	Ra	Shore D	Contact Angle (°)
0%	0.177	89.5	70.47
1%	0.166	89.6	104.7
2%	0.176	85.2	101.46
4%	0.178	70.9	102.01
6%	0.157	61.3	107.93
8%	0.195	63.7	117.45
10%	0.245	69.4	112.5

4. CONCLUSION

The findings of the hydrophobicity and hardness assessment of composites created by incorporating CS particles in varying proportions into epoxy resin are detailed below:

- The size range of CS particles used in the study is narrow, at around 6 µm. The homogeneous size distribution shows that CS particles are suitable for homogeneous dispersion in the epoxy matrix.
- Pure epoxy exhibited a hydrophilic property with a contact angle of approximately 70.5°. With the addition of CS, the water contact angles of epoxy composites increased significantly, and maximum hydrophobic properties were obtained (117.5° with 8% CS). However, a slight decrease in the contact angle (112.5°) was observed with the addition of CS at higher rates (10% CS), which is thought to be due to the agglomeration of particles and the pores formed in the epoxy matrix.

- Low rate (1%) CS additive did not create a visible difference in the hardness of the epoxy. However, when 2% and higher rates of CS were added, a significant decrease was experienced in the hardness values, especially at 6%, where hardness decreased to 61.3. This situation is attributed to the difficulty of the homogeneous distribution of CS particles in the matrix at high rates and the decrease in mechanical performance caused by agglomeration.

The results of this study reveal that calcium stearate additives should be kept at an optimum level in epoxy matrices. It is recommended to use CS at low rates, such as 1-2%, to increase both hydrophobic properties and preserve mechanical properties. The use of higher rates should be evaluated carefully, as they may have negative effects on mechanical properties

5. ACKNOWLEDGEMENTS

6. CONFLICT OF INTEREST

Authors approve that to the best of their knowledge, there is not any conflict of interest or common interest with an institution/organization or a person that may affect the review process of the paper.

7. AUTHOR CONTRIBUTION

İsmail Sinan ATLI contributed to determining the concept and/or design process of the research, managing the concept and/or design process, data collection, data analysis and interpretation of the results, preparation of the manuscript, critical analysis of the intellectual content, and final approval and full responsibility. Hakan ÇİFTÇİ contributed to data collection, data analysis and interpretation of the results, critical analysis of the intellectual content, and final approval and full responsibility.

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