

Manufacture Dry Porous Carbonate Materials with Crack Free

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Abstract pickering foam is a water foam stabilized using solid particles, and this foam can be used as a material in the production of lightweight porous insulating materials. This is done by pouring it into molds, then drying and finally foaming it. One of the most important problems accompanying the drying of bicarbonate foam is the phenomenon of shrinkage in the models resulting from the liquid leaving it by evaporation, resulting in capillary pressures generated inside it. Due to the shrinkage phenomenon, defects and cracks sometimes occur during the drying process that appear on the surface. The process of defects and cracks formation during the drying process depends on the drying conditions such as temperature, relative humidity, and the type of used support angle θ (in addition to the mechanical specifications of the models). Understanding the drying procedures helps researchers and workers in the production of such porous materials, and in choosing the appropriate drying conditions and avoiding any defects in the product. In this study, the effect of the mechanical specifications of the model was investigated by adding surface tension-reducing agents at different concentrations to the models during the drying process, as well as studying the effect of the contact angle (θ) of the support on the probability of crack formation and the mechanical specifications of the dried model. It was found that the value of the contact angle (θ) has a direct effect on cracks and defects. Increasing the value of the contact angle from $\theta = 30^\circ$ to $\theta = 150^\circ$ leads to a reduction or treatment of the cracks that appear in the models. It was also found that adding surface tension-reducing agents such as SLES and SDS leads to crack formation in the models, and increasing the concentration of surface tension-reducing agents such as SLES and SDS leads to foam collapse. However, adding surface tension-reducing agents such as C8AC and C10AC did not show any cracks for all concentrations. As for the mechanical properties of the dry models, they are directly affected by the type of surfactant (C10AC and C8AC), which led to the formation of models with porosities higher than 90%. As for the concentration of the surfactant, it has an effect on the wet model, while it does not affect 90%. From the mechanical specifications (porosity and density) of the model after drying.

Keywords: Pickering foam, Drying, Contact angle, Crack

1 Introduction

Drying is one of the best and oldest methods used by humans for preserving food throughout history. Drying plays a crucial and important role in the production of dried foods, which are essential in supplying food

ingredients. The main advantage of the drying process is to reduce the moisture content in food to prevent spoilage and contamination by microorganisms[1]. Drying can be considered a complex process as it involves the transfer of both heat and moisture. Heat transfer occurs from the air surrounding the material to the material, while moisture transfer occurs in the opposite direction, meaning that heat is transferred from the material being dried to the surrounding air. Therefore, the drying process depends on the surrounding conditions of the material being dried, such as temperature, the speed of the air surrounding the material, and the moisture content of the air[2]. The drying process also plays a role in the production of most building materials, such as porous materials. One of the modern methods used in producing lightweight insulating porous materials is the use of the pickering foam technology.

Foams are dynamically unstable systems, which can be stabilized by adding surface-active agents and solid particles. The demand for the use of pickering foam in the production of porous materials has increased due to its mechanical properties such as porosity, high density, and relative density [3]. Foam can be defined as a water-based foam stabilized by solid particles. It can also be defined as a collection of air bubbles surrounded by liquid, with solid materials located between the bubbles and the liquid, forming a shell around the bubble. There are many solid materials that can be used to obtain stable foam (pickering foam) [4]. Materials such as polymers and some types of powders, such as calcium carbonate (ceramics), were selected in this study. The mechanical properties of pickering foam depend on many factors, including the concentration of the surface tension-reducing substance, the type, size, and temperature and humidity of the solid particles used. pickering foam has many applications, including in the medical, food, and pharmaceutical industries. It is also used in oil and gas refining processes and in the production of modern building materials (insulating materials) [5,6, 7,8,9]

Calcium carbonate is one of the most abundant materials on Earth, as it is available in the form of chalk rocks in

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addition to limestone (which can reach up to 10% of its composition) [10]. Calcium carbonate has important properties that make it suitable for many applications, including its high melting point, pure white color, low solubility in water, and availability at a low cost due to its abundance on Earth. It is also lightweight, resistant to deformation, and provides good thermal and electrical insulation [11, 12].

The cracks and defects are considered among the most important problems associated with the production of porous materials, especially when using the pickering foam technique. Extensive studies and research have been conducted on the spread of cracks and defects to understand the mechanism of their formation. One of the most important causes of cracks and defects in the models to be dried is the phenomenon of shrinkage that occurs during the drying process as a result of the evaporation of the liquid from the model to be dried. The capillary pressure resulting from the shrinkage, which is formed between the air-liquid interface, is the source of internal pressures that cause cracks. In addition, the elasticity modulus of the particles, which determines their response to pressure and the formation of cracks, is also important. The elasticity modulus of the particles may limit the formation of cracks[13]. The properties of the phase of pickering foam are affected by the formation of cracks. Many studies have been conducted on how cracks form in pickering foam, but the studies and research aimed at solving the problem of cracks are very limited[6, 17, 18]. The shrinkage process can result in cracks (defects) that make the foam structure unusable. Therefore, the development of mechanisms through which drying can be controlled, as well as preventing or treating cracks, is essential for producing solid porous structures using particles.

2 Martial and Methods

Two types of materials were used in this study for the purpose of forming a Pickering foam. The first is a solid material which is calcium carbonate (CaCO_3), and the second is liquid materials (DI-WATER) and surfactants.

The solid material: is one of the essential materials used in the formation of Pickering foam. Calcium carbonate (CaCO_3) was used as the solid material in the study, which is available in the local market as a powder. The density of calcium carbonate is 2.70 g/cm^3 , its melting point is 825 degrees Celsius, and the contact angle (θ) of calcium carbonate particles ranges between 10 to 15 degrees[14,17]. The contact angle of this material is very low; therefore, some surfactant chemicals will be added at different concentrations to increase the contact angle between the solid material and the liquid. It is worth mentioning that the best contact angle between the solid material and water is close to 90 degrees to form the best stable foam.

The liquid material: is one of the components of Pickering foam, where deionized water (DI-WATER) was used in this study. It is pure water from which all mineral ions such as calcium, copper, chloride, and others have been removed. This type of water is commonly used in industry, scientific laboratories, and medical applications[18]. The density of deionized water is 1 gram/cm^3 . It does not contain any toxic or harmful substances, and it is also clean. Deionized water is commonly used for cleaning purposes due to its lack of ion concentrations. For the purpose of pouring and studying samples, a plastic (PVC) mold in the form of a disk with a radius of 1.72 cm and a thickness of 0.66 cm was used.

3.Surfactants

Four types of surfactant chemicals were used in three different concentrations in this study. Many surfactant chemicals were used in this study for the purpose of obtaining a foam that can be poured into molds and studied.

- 1- Sodium lauryl ether sulfate (SLES): SLES is widely used as a surfactant in the production of detergents. It is a mixture of primary alkyl sulfates found in many industries, including personal care products[19].
- 2- Sodium dodecyl sulfate (SDS): SDS is also a highly effective surfactant that comes in the form of powder and granules. It has many applications, including car cleaning, due to its high ability to form foam[20].
- 3- Octanoic acid (C8AC): C8AC is a saturated fatty acid that is important for many products and is oily and colorless. It is one of the most promising fatty acids for the production of many industrial products[21].
- 4- Decanoic acid (C10AC): C10AC is a saturated fatty acid used in the production of solvents and rubber[22]. It has properties such as low heat transfer, high latent heat, and good chemical and thermal stability[23].

4.Substrates

Two types of substrates were used as follows:

Hydrophilic substrate: It is a smooth surface that attracts water and has a high surface energy, allowing for surface wetting, and the contact angle is close to or equal to 30 degrees. Its interaction

with water and polar materials is more suitable, from a thermodynamic perspective, than its interaction with water-repellent solvents, as shown in the figure 1 [24].

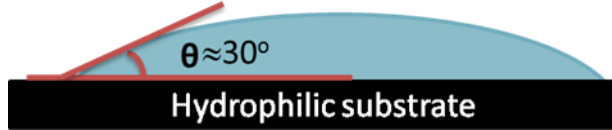


Fig. 1 A hydrophilic surface with a contact angle of $\theta \approx 30^\circ$.

Super hydrophobic substrate. This is also known as a "water-repellent substrate" and is a highly water-resistant substrate that possesses properties such as self-cleaning, water-repelling or stopping, and easy rolling on the surface. The reason for this is due to the fine/nanostructure in addition to the decreased surface energy, and improved performance of heat transfer process. The contact angle is more than 150° as shown in figure 2. Surface roughness also leads to a Super hydrophobic substrate.

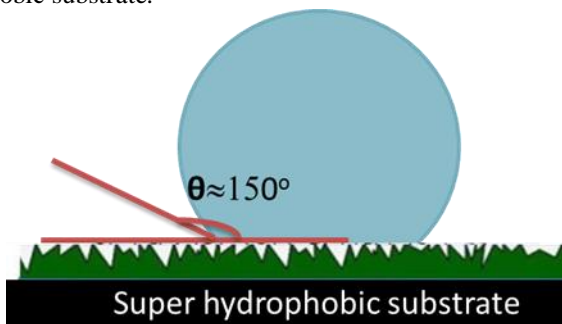


Fig. 2 Super hydrophobic with a contact angle of $\theta \approx 150^\circ$

5. Preparing the sample

At first, both the mold and the base are cleaned with distilled water and then with ethanol. They are dried using nitrogen gas. Baker's foam is prepared by mixing calcium carbonate with ion-free water and one of the surfactants (SLES, SDS, C8AC, C10AC) that reduce surface tension. The mixture is placed in a laboratory glass bottle and a plastic cover (parafilm) is secured on the bottle's nozzle to prevent material leakage during mixing. The glass bottle is placed inside an ultrasonic mixing device that mixes the materials inside the bottle for 10 minutes. The glass bottle is manually shaken for 5 minutes to form foam. Baker's foam is poured into the mold, which is fixed on the base. The model is adjusted using an iron blade to obtain the desired shape. Finally, the mold is lifted and placed inside a testing device.

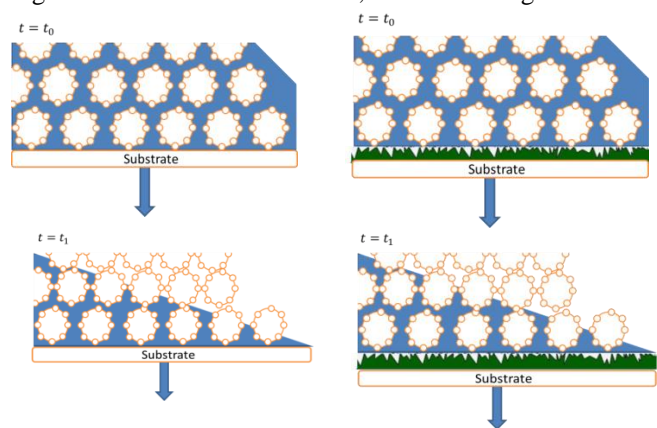
6. Testing device

The testing device consists of a transparent square box with dimensions of (180,170,150 mm). A

thermometer and a humidity gauge are placed inside the box to record the temperature and humidity inside the box during the drying process. It also includes a laboratory scale to measure the sample weight during the drying process, and it is placed around the silica gel model to obtain a stable temperature and humidity. The sample is monitored by a laboratory camera that takes pictures of the model's surface at certain intervals. A pointer is used to capture a side view of the model.

7. The Cracks

This is a phenomenon that occurs in models during the drying process due to the shrinkage phenomenon that occurs as a result of water loss through evaporation[25]. The formation of cracks depends on the drying conditions (temperature and humidity) and the mechanical properties of the models. The tensile stresses that occur as a result of shrinkage inside the models are responsible for the formation and appearance of cracks[4]. The models to be dried undergo shrinkage and contraction, and the addition of some porous materials during the drying process. The shrinkage process leads to the formation of cracks over time. Therefore, the forces that affect the formation of cracks include adhesion force and cohesion force, and the force that overcomes them. The adhesion force is between the sample particles and the support, while the cohesion force is between the sample particles. These forces prevent the samples from shrinking during the drying process (resistance to stress). The value of the adhesive force depends on the contact area between the support and the sample, while the cohesive force is an internal property of the sample particles. As a result of these forces, cracks are formed because the adhesive force is greater than the cohesive force, as shown in figure 3.



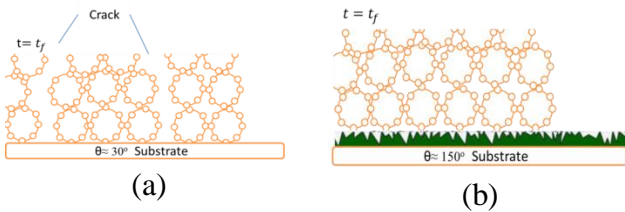


Fig. 3 shows the effect of the support on the pickering foam. (a) A hydrophilic support has a high adhesive strength and the adhesion force is greater than the cohesive force. (b) Super hydrophobic support has a low adhesive strength and the adhesion force is less than the cohesive force

The effect of the contact angle (θ): Figure 4 illustrates the drying process of a model to which a surfactant (SDS) has been added. It was poured onto two supports with different contact angles, one hydrophilic and the other hydrophobic. During the drying process, cracks appear in the model on the hydrophilic support due to the increased wettability of the support and its high surface energy (high adhesive strength), which, in turn, cannot alleviate the stresses resulting from capillary forces, leading to cracks. On the other hand, there are no cracks in the model on the hydrophobic support because the support has a low energy and can alleviate the stresses caused by capillary forces during dryin

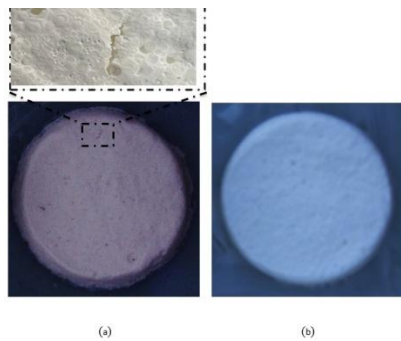


Fig. 4 The effect of contact angle on crack formation behavior in models using a surfactant (0.17% SDS). (a) Contact angle of approximately 30 degrees. (b) Contact angle of approximately 150 degrees

Effect of Surfactants: Figure 5 illustrates the effect of adding different surfactants (SLES, SDS, C8AC, C10AC) at certain equal ratios on the probability of crack formation. The models were poured onto a hydrophilic substrate ($\theta \approx 30^\circ$) under monitored drying conditions. The addition of surfactants to the mixture (calcium carbonate and deionized water) changed the properties of the models (Bickernack foam), including the contact angle between particles. At the beginning of the drying process, the SLES model started cracking in its center, then moved towards its edges, and the cracks increased until the model dried. On the other hand, we observed that the SDS surfactant evaporated with the liquid and the sample shrank, and with continued drying,

cracks appeared in the model at lower ratios than adding SLES. As for the two surfactants C8AC and C10AC, we observed no cracks on their surface during the drying process

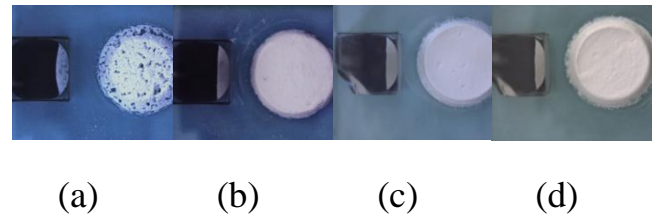


Fig. 5 The effect of surfactants on the formation of cracks in models during the drying process, with certain equal ratios of (SLES, SDS, C8AC, C10AC). The models were poured on a hydrophilic substrate ($\theta \approx 30^\circ$) (a)SLES (b) SDS(c) C8AC(d) C10AC

Effect of concentration of surfactant materials: Figure 6 illustrates a sequential process for the effect of increasing concentrations of surfactant materials on the model (calcium carbonate and ion-free water) in the likelihood of cracking. Three concentrations (1.97, 0.47, and 0.26)% of surfactant material (SLES) were used. A water-loving substrate ($\theta \approx 30^\circ$) was poured onto the models. Model (a) shows no cracks at the beginning of the drying process, but cracks begin to appear over time until the model dries out. As for concentrations (b) and (c), we observe non-cohesive foam and large bubbles when lifting the mold, and as the drying process continues, the model collapses as the bubbles merge with each other.

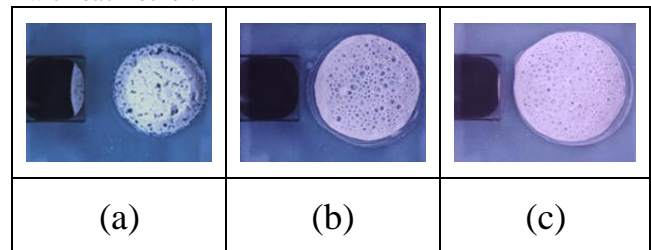


Fig. 6 The effect of concentration of surfactant materials (SLES) on crack formation in models during the drying process is demonstrated on a substrate with an approximate angle of 30 degrees ($\theta \approx 30^\circ$). Concentrations (c) 1.97%, (b) 0.47%, and (a)

8. Change in Sample Dimensions:

The change that occurs in the dimensions of the sample during the drying process due to the shrinkage caused by capillary pressures resulting from liquid evaporation. The change in dimensions depends on the percentage of liquid that exits the sample through evaporation during drying. If all the liquid in the sample evaporates and is replaced by air, there will be no shrinkage during the drying process and the dimensions

will remain constant. However, if the volume of some liquid molecules is replaced by air, shrinkage occurs. The change in dimensions of the sample during drying is shown using a laboratory camera and through data collected such as the diameter and thickness to calculate the sample volume.

In this part of the chapter, we examine the models in which no cracks occurred when adding octanoic acid (C8AC) or decanoic acid (C10AC) to a mixture of calcium carbonate and

deionized water. No cracks were observed at all concentrations when poured on a substrate with a contact angle of 30° and 150°. The deformation that occurs in the sample during the drying process leads to a change in the dimensions of the model. The shrinkage process begins as a result of the compression of all particles in the Baker's yeast. Thus, the particles acquire sufficient forces to resist the capillary forces.

9. Change in Sample Volume:

Figure 7 illustrates the change in volume of a sample in which no cracks occurred (c8ac), fixed on a water-repellent substrate ($\theta \approx 150^\circ$), for three concentrations. From the figure, it can be observed that the volume of the sample changes (decreases) during the drying process. The final volume change was found to be (45%) at a concentration of 0.08%, (49%) at a concentration of 0.16%, and (50%) at a concentration of 0.68%.

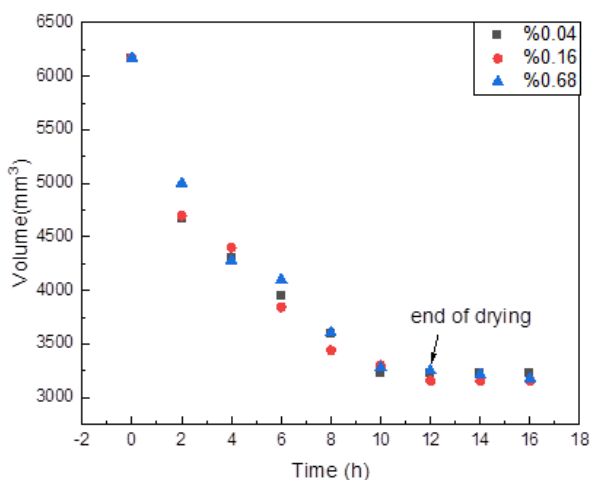


Fig. 7 Volume change during drying process for samples with three concentrations (0.68%, 0.16%, and 0.08%) of (c8ac) on a Super hydrophobic substrate at an angle of $\theta \approx 150^\circ$

Porosity: Porosity has an effect on the mechanical properties of porous materials used in engineering applications. Porosity is defined as the ratio of the volume of air inside the sample to the total volume of the sample. The porosity of the sample depends on the

amount of air that is replaced by the liquid inside the sample and the degree of shrinkage inside the sample during drying. Figure 8 shows the changes in porosity during the drying process of the sample for three concentrations (0.68, 0.16, and 0.08)% of the (c8ac) material fixed on a Super hydrophobic substrate at $\theta \approx 150^\circ$. The pores gradually appear and we observe a linear increase in the porosity of the sample, which means that the change in the volume of air inside the sample is equal to the change in the sample volume. Then, the porosity stabilizes due to the shrinkage of the sample, i.e. the change in sample volume is greater than the volume of air, and the porosity remains constant until the end of the drying process. When the porosity is a fractional addition of (c8ac 0.08%) higher than the porosity at concentrations of 0.16% and 0.68%, and in addition to a porosity of 0.16%, it is slightly higher than 0.04%.

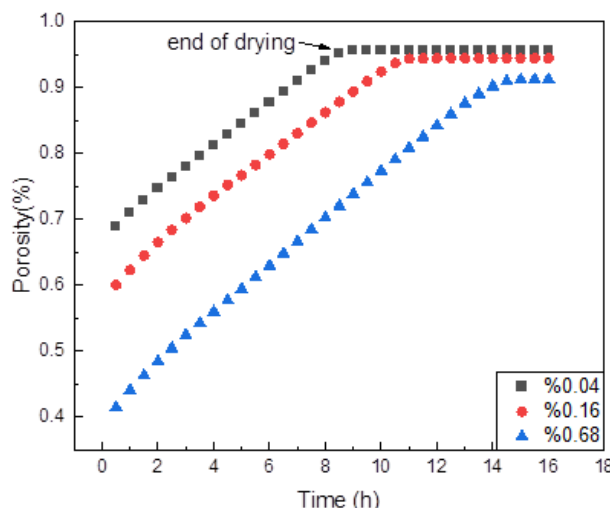


Fig. 8 Change in porosity during the drying process of models for three concentrations (0.68, 0.16, and 0.08)% of (c8ac) material fixed on a Super hydrophobic substrate $\theta \approx 150^\circ$

Density is divided into two types: the first is apparent density, also known as volumetric density, and the second is relative density. Apparent density (bulk density) is defined as the mass of the sample divided by its volume Figure 9 illustrates the change in apparent density during the drying process for samples of three concentrations of (c8ac) fixed on Super hydrophobic substrate at $\theta \approx 150^\circ$. It is observed that the apparent density continuously decreases with time, and the best apparent density obtained for the sample is 0.04%, followed by 0.16%, and then 0.68%. The evaporation process of water in the samples keeps distances between particles of calcium carbonate, which leads to a decrease in its density, and consequently, the particles become smaller in size, in addition to reducing the distance between particles.

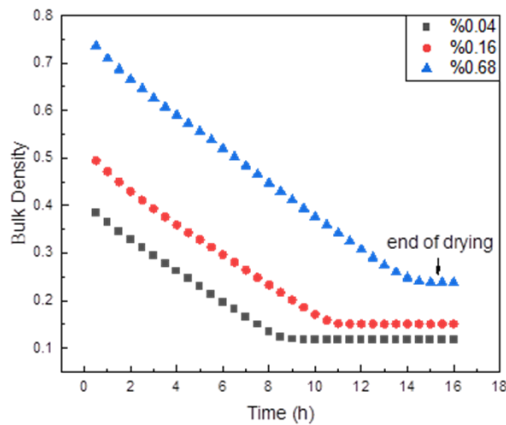


Fig. 9 Change in apparent density during the drying process for samples of three concentrations (0.68%, 0.16%, and 0.08%) of (c8ac) fixed on Super hydrophobic substrate at $\theta \approx 150^\circ$

Relative density is defined as the ratio of apparent density to the density of the solid material (calcium carbonate), and it can be calculated using this equation. Figure 10 illustrates the change in relative density during the drying process for samples with three different concentrations of (c8ac) fixed on a Super hydrophobic substrate at $\theta \approx 150^\circ$. It is observed that the relative density decreases with time during the drying process, and the best relative density obtained for the sample is at a concentration of 0.04%, followed by 0.16%, and then 0.68%.

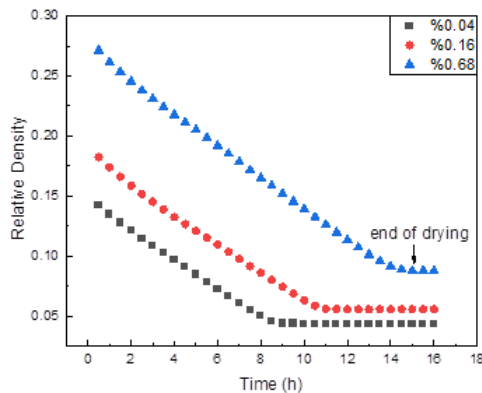


Fig. 10 Change in relative density during the drying process for samples of three concentrations (0.68%, 0.16%, and 0.08%) of (c8ac) fixed on a Super hydrophobic substrate at $\theta \approx 150^\circ$

9. Conclusion

The formation of cracks in the models during the drying process depends on the type and concentration of the surfactant used. The use of (C8AC, C10AC) surfactants at all concentrations contributes to the production of foam free of cracks and defects. The concentration of the surfactant does not have a clear effect on the mechanical properties of the crack-free and defect-free dry models.

On the other hand, it has been observed that the concentration of the surfactant has an effect on the mechanical specifications of the wet models (before the drying process). The angle of the substrate has a direct and clear effect on the formation of the models during the drying process. An angle of $\theta \approx 30^\circ$ to $\theta \approx 150^\circ$ contributed significantly to reducing and treating cracks and defects.

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