



Effect of Dipping Time of Filter Paper using SiO₂-Chitosan Nanocomposite on Contact Angle for Water and Oil Separation

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Abstract: Wastewater enriched with organic compounds into the aquatic environment can cause a decrease in dissolved oxygen, which significantly affects microorganisms living in this environment. Ultimately, this problem endangers the survival of organisms at higher trophic levels. Recently, the development of membrane technology has attracted significant attention, as membranes are increasingly used in the separation of oily wastewater. Membrane technology has been shown to be effective in removing oil from water. An effective membrane for this purpose is a hydrophobic silica composite membrane, which is synthesized using cellulose paper substrate and silica derived from rice husk to create the active membrane layer. In one part of the current investigation, the impact of staining duration was evaluated by varying the soaking time for SiO₂-chitosan nanocomposite-coated filter paper at 5, 10, 15, and 20 min. In this study, the contact angles for immersion times of 5, 10, 15, and 20 min were measured and recorded at 94.35°, 98.53°, 101.04°, and 104.34°, respectively. In addition, the effectiveness of water-oil separation was evaluated using cellulose paper coated with SiO₂/chitosan composite, with efficiencies of 80%, 85%, 87.5%, and 90% recorded for this process.

Keywords: Cellulose Paper; SiO₂; Chitosan; Contact Angle; Hydrophobic.



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1. Introduction

The presence of oily wastewater, containing high concentrations of organic compounds, can cause a decrease in oxygen levels, threatening microorganisms living in it. As a result, such environments have a detrimental impact on the viability of higher trophic levels[1]. In such a scenario, oxygen plays a critical role; in an environment with low oxygen, both chemical and biochemical processes often produce byproducts, resulting in undesirable color, taste, and odor in the water[2]. Additionally, water-oil emulsions add another layer of complexity to the effective treatment of such polluted water. The efficient treatment of oil-contaminated water is hindered by the formation of water-oil emulsions. A significant source of oily wastewater is the improper

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disposal of household cooking oils [3]. Often, residues of cooking oils are thrown down the drain, and this contaminated water eventually reaches rivers and oceans. Improper disposal in this way can cause extreme pollution, forming a thin layer on the water's surface, blocking natural photosynthesis, and destroying habitats for aquatic flora and fauna [4]. Thus, in efforts to improve water purity and ensure its safe use, it is essential to implement processes that minimize water pollution. Several studies have been conducted to treat oily wastewater using a variety of techniques, including the use of membrane technology with cellulose paper[5].

Cellulose paper is a cellulose-fiber material composed of compounds that are insoluble in water. Its inherent insolubility in water characterizes cellulose paper, a renewable material widely used in the pulp and paper industry [6]. Despite cellulose's naturally low absorption capacity, its absorption properties can be enhanced through various modifications [7]. Hydrophobic aerogels can be synthesized from cellulose nanofibers derived from wood or agricultural residues. When modified, cellulose paper can be engineered to repel water and absorb oils by incorporating hydrophobic materials [8].

The term 'hydrophobic' originates from the Greek words 'hydro' (water) and 'phobia' (fear), describing a property that prevents the absorption and adhesion of water. Hydrophobic materials include compounds that do not dissolve in water but are soluble in oils [9]. The mechanism of hydrophobicity is best illustrated by the behavior of a lotus leaf, a property resulting from the combination of micro- and nanostructures on the leaf's surface, which create an air layer between the leaf and water droplets, along with a waxy cuticle that reduces the surface energy of the leaf. When water comes into contact with the surface of a lotus leaf, it forms a high contact angle of about 160° , causing droplets to roll off and carry away particulates such as dirt, thereby keeping the leaf surface clean—a phenomenon known as the self-cleaning effect [10].



Figure 1. Water Droplets on Lotus Leaves

Hydrophobicity is defined as the property of repelling water, and it is best demonstrated by a contact angle between 90° and 150° . A contact angle greater than 150° indicates superhydrophobicity. Measuring the contact angle on a specific substrate is a key method for assessing hydrophobic properties. Based on the contact angle, the surface can be classified into several categories: a contact angle below 90° indicates superhydrophilicity; a contact angle between 90° and 150° indicates hydrophobicity; and a contact angle above 150° indicates

superhydrophobicity. Additionally, a contact angle approaching 0° signifies extreme superhydrophilicity [11].

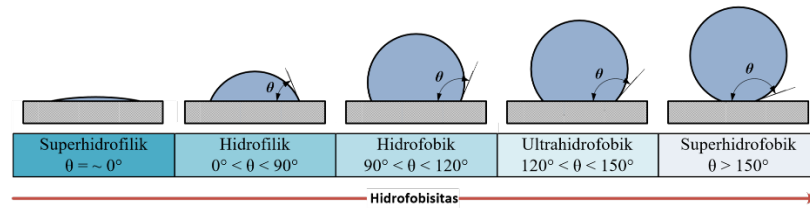


Figure 2. (a). superhidrofilics (b). hidrofilics (c). hidrofobic (d). ultrahidrofobics (e). superhidrofobic

Research has been conducted on developing superhydrophobic coatings inspired by coral structures, aimed at improving the filtration of water and oil using filter paper. In this study, a drop-coating technique was used to deposit a modified silica/polystyrene composite coating onto filter paper, imparting superhydrophobic properties to its surface. The silica particles used in this study were synthesized via the Stober process [12]. To ensure high-quality output, the reaction duration, preparation technique, and the concentration of NH_4OH in the silica particle synthesis were carefully monitored, both in the synthesized silica particles and in subsequent coating and characterization processes. Additionally, auxiliary materials must be incorporated during synthesis to ensure high-quality output [13]. One such material is chitosan, a naturally derived biopolymer containing amino and hydroxyl groups in its structure, making it one of the most effective immobilization matrices with properties such as membrane formability, effective adhesion, cost-effectiveness, non-toxicity, high mechanical durability, high hydrophilicity, and the ability to stabilize the system [14].

Due to the hydroxyl groups present in its structure, chitosan is inherently highly hydrophilic. Introducing hydrophobic properties to chitosan, however, can be achieved by modifying the chemical structure of the chitosan polymer chain. One of the most common methods is to incorporate hydrophobic groups into the chitosan molecule. For example, alkyl groups such as methyl, ethyl, or propyl can be attached to the amino groups of the chitosan polymer chain. Building on previous research, this study investigates the "Effect of Dipping Time of Filter Paper Using SiO_2 /Chitosan Nanocomposite for Water and Oil Separation". This research aims to enable efficient and cost-effective separation of oily liquid waste with high filtration performance, achieving hydrophobic surface contact angles $>90^\circ$ on the substrate (cellulose paper) [15].

2. Materials and Method

This study is an experimental investigation of the impact of varying dipping times of SiO_2 -chitosan nanocomposite-treated filter paper on contact angles for water and oil separation. Experiments were conducted in May 2024 at the Physics and Chemistry Department Laboratories, Faculty of Mathematics and Natural Sciences, University of Negeri Padang. First, the filter paper was coated with a SiO_2 -chitosan solution. Following the coating process, contact angles were measured. The contact angle tests involved measuring contact angles by dropping water droplets from a height of 2 cm onto a cellulose paper substrate coated with a SiO_2 -chitosan solution using a syringe. Frontal images of water droplets on the substrate were captured using a DSLR camera,

with careful adjustments to contrast, lighting, and focus [16]. Contact angles were measured using specialized software and a series of camera setups, as illustrated in Figure 3.

The measurement of contact angles utilized software in an attempt to counteract any errors in measurement through a kind of camera apparatuses in sequence for contact angles and a diagram representing apparatuses utilized, such as in Figure 3.

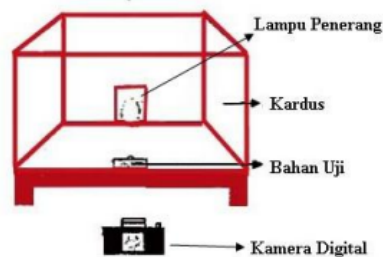


Figure 3. Schematic of the contact angle photographing device

Figure 3 is a schematic diagram of the instrument setup for measuring contact angles. This setup includes a cardboard frame with openings at both ends, covered with tissue at the rear opening to prevent direct light exposure to the sample. The DSLR camera, sample, and light source are aligned linearly in a single plane, allowing an image to be captured that displays the contact angle. Sequentially, water droplets are ejected from a height of 2 cm onto the sample surface using a syringe, and an image of the sample surface is captured to assess its hydrophobicity. During the measurement process, the shape of the droplet on the sample surface is observed, and the contact angle is measured [17] .

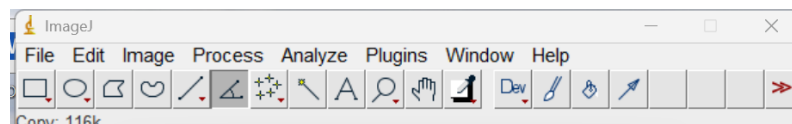


Figure 4. ImageJ software view

To establish a contact angle, a line is drawn between the droplet and the sample's surface, creating an angle between them. Initially, it is achieved through choosing "the angle tool" in Figure 4, whose function is to measure the present angle in a droplet.

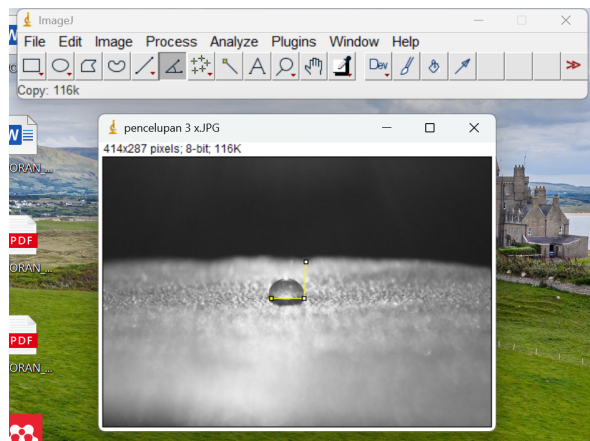


Figure 5. Angle measurement on droplets using ImageJ

Subsequently, draw a yellow line that forms an angle with the droplet. Next, select 'Measure' and then 'Analyze'. Additionally, after several measurement repetitions, the measured values are analyzed to determine the average. Multiple measurement repetitions are performed to ensure high accuracy.

3. Results and Discussion

The following photos of contact angles from testing each sample with variations in immersion time (5, 10, 15 and 20 minutes) can be seen in the following figure:

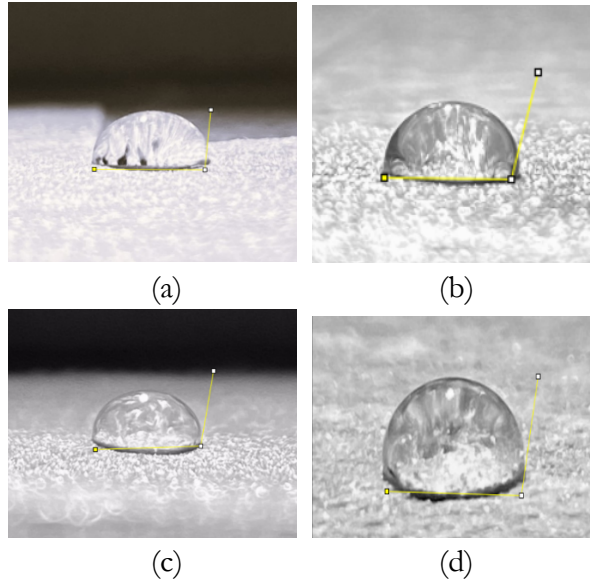


Figure 6. angle contact with variation (a). 5 minutes (b). 10 minutes (c). 15 minutes (d). 20 minutes

In this study, contact angles were measured using processed data analyzed with ImageJ software. The primary objective was to measure contact angles to determine the optimal immersion time for creating an efficient hydrophobic layer. A surface is considered hydrophobic when the contact angle exceeds 90° . In this work, the contact angle for a 20-minute immersion was compared with those for four other immersion times, as shown in Figure 7:

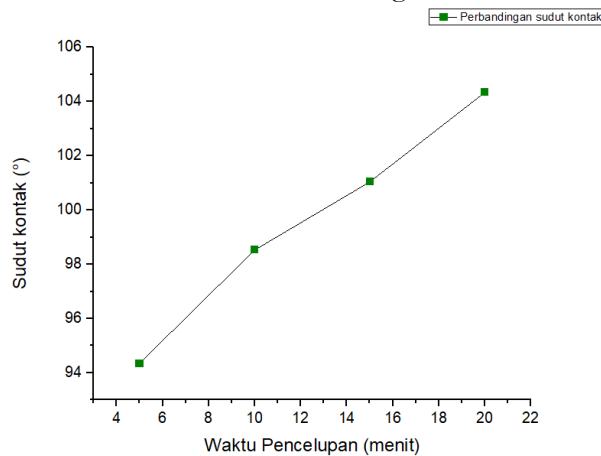


Figure 7. Contact angle comparison chart

The graph demonstrates that the variation in the dipping time of cellulose paper with SiO₂/chitosan composite solution affects the resulting contact angle. In this study, four different dyeing times were tested: specifically, 5, 10, 15, and 20 minutes. This study employed the dip-coating method, performed manually. The dipping process involved immersing cellulose paper in the SiO₂/chitosan composite solution for 5 minutes, removing it and allowing excess solution to drip off, and then placing it in an oven at 100°C for 15 minutes. This process was repeated for each specified immersion time. Contact angles were calculated using ImageJ software by measuring each sample three times to ensure accuracy, and the results were averaged. The average contact angles for each immersion time are presented below.

Table 1. Measurement results of each contact angle

Dipping Time	Contact Angle
5 mintues	94.35°
10 minutes	98,52°
15 mintues	101,04°
20 minutes	104,34°

4. Conclusion

The contact angles measured for all samples in relation to immersion times are 94.35°, 98.52°, 101.04°, and 104.34°, respectively. There is a positive relation between contact angles measured and increased immersion times, signifying hydrophobicity. Measured contact angles are evaluated in an attempt to detect an ideal duration for immersing a substrate for a hydrophobic layer to form. For a surface to be considered hydrophobic, a contact angle must be greater than 90°. In this work, a contact angle corresponds to an 20-minute period of immersion.

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