

The Effect of Applied Vibrations on Structural Features and Wettability of Nanopores of Anodic Alumina Oxide Formed by Two-step Anodization Process



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#### **INTRODUCTION**

Due to its well-ordered structure, large surface area, and ability to control the diameter of nanopores, porous anodic aluminium oxide (AAO) continues to attract the attention of scientists [1,2]. AAO pores have adjustable sizes that range from a few to hundreds of nanometers.

In addition to the fundamental research community, the wettability of solid surfaces has practical implications in fields such as micro/nanofluidic technology and microelectronics [3]. Moreover, the wettability of the porous AAO surface can be adjusted between hydrophilic and hydrophobic without any chemical modification [4]. By controlling the diameter of the pores, the wetting angle can be controlled. In this way, it is possible to obtain surfaces of various wettability, which would be applicable in different areas.

To create a template with reproducible characteristics that could be used to fabricate a variety of membranes and structures, it is important to study the surface wettability of AAO. Therefore, in this study, the results of the investigation of the production and wettability properties of AAO with different pore sizes were presented. Furthermore, this study investigated how the wettability of AAO changes due to vibration application during the anodization process. As a result, AAO samples with various morphological and wettability characteristics were obtained.





### **ANODIZATION PROCESS**



Fig. 1. Mechanism of anodization technique



# A NOVEL DESIGN OF ELECTROCHEMICAL REACTOR



**Fig. 2.** 3D model of the electrochemical reactor for the fabrication of nanoporous AAO membranes

## **TWO-STEP ANODIZATION**



The first step of anodization (1 h)

Fig. 3. Experimental setup of the two-step anodization process

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#### WETTABILITY MEASUREMENT



# **Fig. 4.** Illustration of the hydrophobic analysis of the AAO membrane contact angle measurements

### **RESULTS (1)**

AAO specimens were used to measure the contact angle and the pore diameters ranged from 28 to 61 nm.

Parameter	Voltage, V	Temperature, °C			
		5	10	15	
Diameters of pores, nm	40	28 ± 10	33 ± 10	41 ± 10	
Interpore distance, nm		87 ± 10	86 ± 10	85 ± 10	
Porosity, %		9	13	21	
Contact angle, deg		60	66	71	
Diameter of pores, nm	50	38 ± 10	$40 \pm 10$	55 ± 10	
Interpore distance, nm		$105 \pm 10$	$101 \pm 10$	$105 \pm 10$	
Porosity, %		12	14	25	
Contact angle, deg		65	66	76	
Diameter of pores, nm	60	48 ± 10	52 ± 10	61 ± 10	
Interpore distance, nm		$108 \pm 10$	113 ± 10	$110 \pm 10$	
Porosity, %		18	19	28	
Contact angle, deg		68	70	80	

Table 1. The morphologies of AAO membranes with different porosity and contact angle measurements

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### **RESULTS (2)**

Parameter	Pore diameter, nm	Interpore distance, nm	Porosity, %	Contact angle, deg
No excitation	48 ± 10	$108 \pm 10$	18	68
Excitation frequency 0.35 kHz	57 ± 10	117 ± 10	22	74
Excitation frequency 2.03 kHz	86 ± 10	$126 \pm 10$	42	89

Table 2. The morphologies of AAO membranes with different porosity and contact angle measurements

AAO specimens were used to measure the contact angle and the pore diameters ranged from 48 to 86 nm. Without any chemical alteration, a higher water contact angle  $CA = 89^{\circ}$  was achieved on the surface of the porous AAO when the diameter reaches 86 nm. By altering the synthesis conditions, different surface morphologies of such AAO were generated, allowing the surface characteristics to change from hydrophilic to hydrophobic.



#### CONCLUSIONS

According to research, aluminium can be anodized during two-step anodization process with an oxalic acid electrolyte to create AAO membranes. Fabricated AAO membranes with pores ranging in size from 28 to 86 nm. The following conditions were chosen for anodization process: voltages of 40 V, 50 V and 60 V and temperatures of 5 °C, 10 °C and 15 °C in 0.3 M oxalic acid. Also, AAO membranes were investigated when vibrations were used during anodization process and wettability was analysed. The surface chemistry of the AAO membranes was left unchanged to focus on investigating the effect of the AAO surface morphology on the wettability parameters. The water contact angles were measured on the porous AAO surface of different pores diameters. As the diameter increased, the wettability of the porous AAO gradually changed from hydrophilicity to hydrophobicity. The AAO surface morphology affected the contact angle values. With changes in the synthesis conditions, different surface morphologies of such AAO membranes could be created. The results showed that suitable AAO fabrication conditions (temperature, voltage, and vibration) could be selected for future applications of AAO membranes.

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