Categorization of Aluminium Oxide Nanoporous media based on the Pore Circularity using Image Processing Techniques

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Abstract: Morphology of fabricated nano-templates plays a significant role in nanoengineering and technology. The geometrical properties of nanopores in synthesized nanomembrane is influenced by various anodizing parameters such as anodizing time, the concentration of acid, voltage applied and temperature of bath solution. The current study exhibits a computerized, automated tool to extract and measure geometrical features of nanoporous media. Nanopore radius and nanopore circularity of Anodic Aluminium Oxide (AAO) are determined in this study by different anodizing parameters. The experimental results illustrate that as the anodization time is increased (5mins, 9mins, 20mins and 30mins) while keeping the constant value in concentration (5%), temperature (20^0C) and voltage (50 V), the nanopore radius have gradually increased from 7.9nm to 11.6nm and nanopore circularity has decreased from 0.73 to 0.56. Similarly, as the voltage is increased (35V, 40V, 45V), the nanopore radius increased gradually from 8nm to 13.3nm and the nanopore circularity increased initially from 0.68 to 0.78 and decreased to 0.66 by maintaining the concentration (4.7%), time (8min) and temperature (5^0C) constant. When the concentration (4% and 5%) and temperature (20^0C and 25^0C) has been altered keeping time (20 min) and voltage (50 V) constant, the increase in nanopore radius from 55.6nm to 67.4nm and increase in nanopore circularity from 0.55 to 0.67 is observed. The features drawn from values of nanopore radius and circularity are verified by professional chemical experts, which ensure the exhaustiveness of the proposed results.

***IndexTerms* - Anodic Aluminium Oxide, Aluminium Nanopore, Characterization, FESEM, Pore radius, Pore circularity, Nanomaterial, Nanotechnology.**

I. INTRODUCTION

Anodic Aluminium Oxide (AAO)[1] has gained interest among researchers in nanotechnology due to its distinct and evident pore architecture and other properties such as corrosion resistance, thermal stability, insulation and abrasion resistance. As a host material or a nano-template, AAO plays a vital role in numerous surface engineering applications like molecular separation, catalysis, sensors, energy storage, electronics, drug delivery and template syntheses. In several nanostructured materials, it is an important component in the form of nanodots, nanowires and nanotubes[2-6]. Transport mechanism, important flow and predicting flow properties of various porous media relevant to fundamental and industrial applications can be described by pore geometry[10-11]. The advantage of nanotechnology relies upon the way that it is conceivable to tailor the structures of materials at incredibly small scales to accomplish explicit properties, therefore enormously expanding the material science toolkit. Utilizing nanotechnology, materials can viably be made stronger, lighter, durable, more reactive and better electrical conductors. There are numerous every day utilized commercial products in the market based on the nanoscale materials and its process.

In the last couple of decades, many nano researchers have shown interest in nanoscale materials, owing to its importance. The application of nanoporous templates was presented by C. Sousa [7]. Solar desalination through aluminium nanoparticles was studied by L. Zhou [8]. X Yang worked on the topological parameters of nanopore [9]. Computational geometry was studied by Joost H. [12]. The geometrical features of Al_2O_3 nanopore images were proposed by P. Bannigidad and Jalaja U. [13-15].

The main objective of the current study is to extract geometric values of nanopores radius and nanopores circularity from the Al_2O_3 FESEM images using global thresholding method. Transport mechanism and predicting flow properties of various porous media relevant to fundamental and industrial applications are described by the properties extracted out of aluminium nanoporous images using the proposed method.

II. MATERIALS AND METHOD

Aluminium nanoporous FESEM images (A, B, C and D) are obtained at regular intervals of time (5 mins, 9mins, 20 mins and 30 mins), keeping constant the concentration (5%), temperature (20^0C) and voltage (50 V). The images (E, F and G) are obtained at varying voltage (35V, 40V, 45V) keeping the concentration (4.7%), time (8min) and temperature (5^0C) constant. Images (H and I) are obtained at 4% and 5% concentration, 20^0C and 25^0C temperature respectively keeping time (20 mins) and voltage (50V) constant.

III. PROPOSED METHOD

The objective of the present study is to develop an automated tool to determine the effect of changing anodization parameters on the geometrical features; namely, the nanopore radius and nanopore circularity of Al_2O_3 nanopores in the experimental FESEM images. In contrast with the current manual staining techniques the proposed method is more efficient, accurate, reliable and robust. The top views of anodized Al_2O_3 FESEM images are shown in Fig.1. Images labeled A, B, C and D are obtained at regular intervals of time (5 mins, 9 mins, 20 mins and 30 mins), keeping constant the concentration (5%), temperature (20^0C) and voltage (50 V). The images E, F and G are obtained at varying voltage (35V, 40V, 45V) keeping the concentration (4.7%), time (8min) and temperature (5^0C) constant. Image H and I are obtained at 4% and 5% concentration, 20ºC and 25ºC temperature, 20 mins time and 50V voltage.

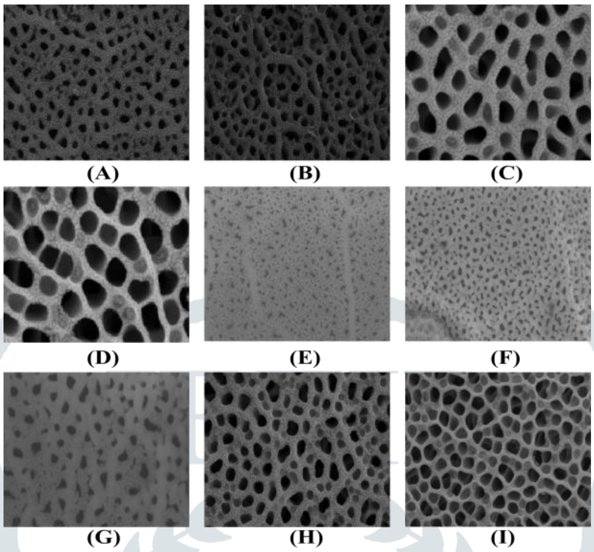

Figure 1. Top view of aluminium oxide FESEM

The geometric features extracted from the images; nanopore radius and nanopore circularity are defined as below:

1. Nanopore radius: Radius is defined as half, the average of nanopore major axis and minor axis.
2. Nanopore circularity: The nanopore circularity is defined by using the below equation:

$$C=(4\pi.S)/L^2$$

Where, S=Surface area occupied by single nanopore.
L= Nanopore perimeter.

The following facts stand true with nanopore circularity (C):

- C = 1, when the nanopore is an ideal circle.
- C < 0, when the nanopore deformation occurs.
- C value is close to 0, when nanopore is similar to elongated polygon.

The algorithm of the proposed method:

1. Read aluminium nanopore FESEM image.
2. Convert the given input image to RGB image using the function rgb2gray()
3. Perform pre-processing operations on step 2:
   a. Applied image enhancement to enhance the quality of the image
   b. To obtain binarized image, the following steps are applied:
```
for  i=1:size(gray_image,1);
 for j=1:size(gray_image,2);
 if gray_image(i,j)>121
       binary_image(i,j)=1;
else
       binary_image(i,j)=0;
end
end
end
```
   c. Perform morphological operations on the binarized image.
4. Segment the individual nanopores from the binarized image using the below method.
```
filteredpours=ones(1,num);
for segno=1:num
 if length(find(L==segno))<100
```

```
        L(find(L==segno))=0;
        filteredpours(segno)=0;
    end
end
```

5. Extract the geometric features; i.e., nanopore radius and nanopore circularity and store them as knowledge base.
6. Finally, analyse and interpret the results using the following conditions:
   If nanopore circularity = 1 then, the nanopore is an ideal circle.
   Else if nanopore circularity < 0, then, the nanopore deformation occurs.
   Else if nanopore circularity value is close to 0, then nanopore is similar to elongated polygon.

The flow diagram of the proposed method is depicted in the below Fig. 2:

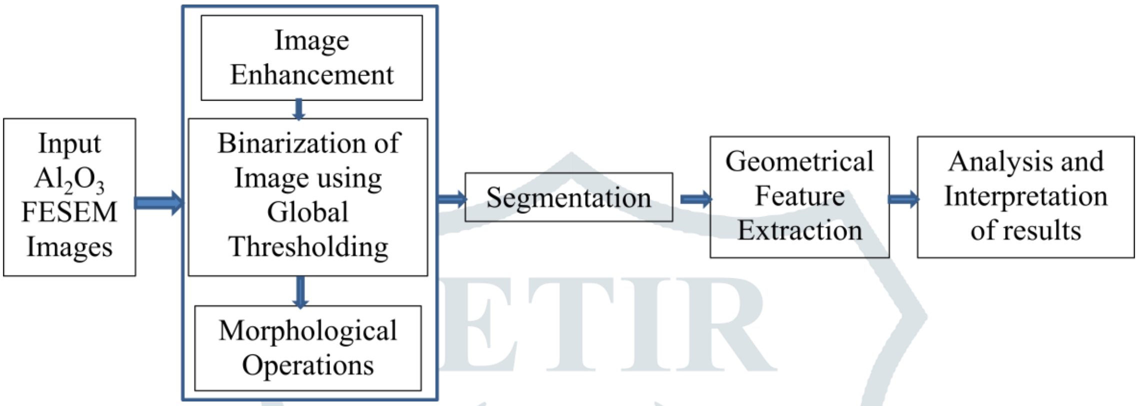

Figure 2. Flow diagram of proposed method

IV. EXPERIMENTAL RESULTS AND DISCUSSION

The experimentation is carried out on Intel(R) Core(TM) Duo T6670 @ 220GHz with 2 GB RAM using MATLAB R2014a software tool. The images used in the experimentation are of 500*500 dimension with 205.24KX magnification and are obtained from the Department of Chemistry, Rani Channamma University, Belagavi at varying anodizing properties. The details of these Al_2O_3 FESEM images are depicted in Fig 1.

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The FESEM images are initially enhanced and then converted to binary image using global thresholding method (Fig2. (ii), Fig. 3 (ii) and Fig. 4(ii)). The binarized images will undergo the morphological operation and then the nanopores are extracted through segmentation (Fig2. (iii), Fig. 3 (iii) and Fig. 4(iii)). The nanopore radius and nanopore circularity is computed for every nanopore. Finally, the results are interpreted, analysed and categorised based on the values of nanopore circularity criteria (discussed in section 2).

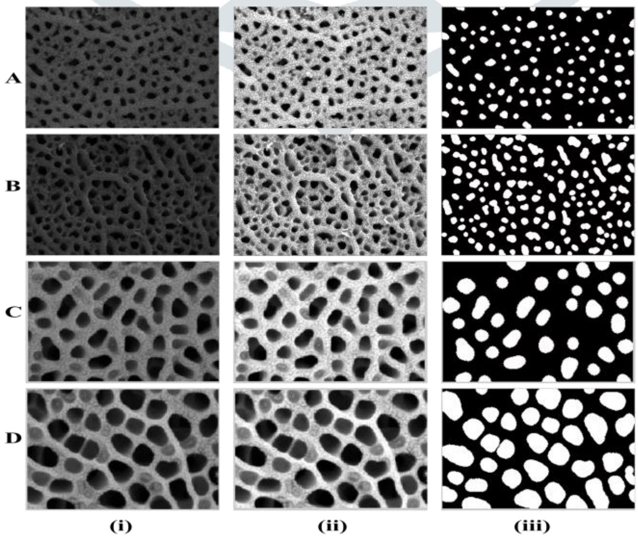


Figure 3. (i) Original FESEM images captured at different intervals of time, (ii) Grayscale images, (iii) Segmented images

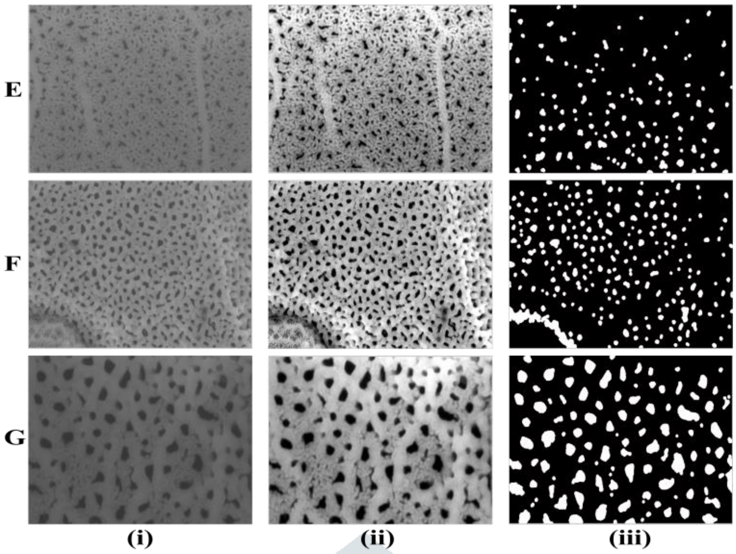


Figure 4. **(i) Original FESEM images captured at different voltage, (ii) Grayscale images, (iii) Segmented images**

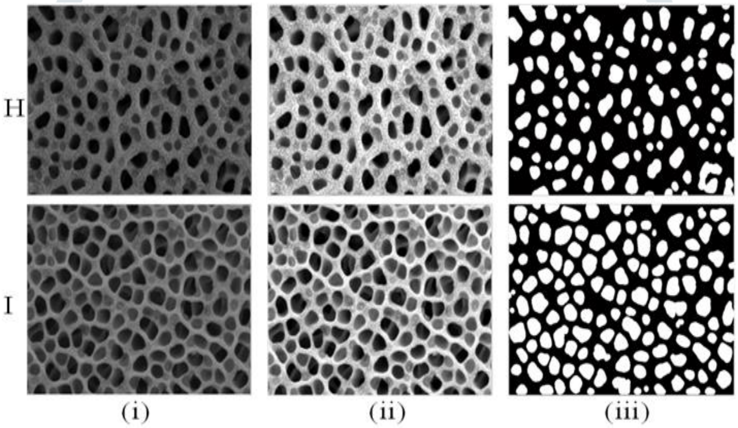


Figure 5. **(i) Original FESEM images captured at different temperature and concentration, (ii) Grayscale images, (iii) Segmented images**

The average results of Al_2O_3 nanopore images categorised based on nanopore circularity is shown in the Table 1.

Table 1. The average results of aluminium oxide nanopore images categorised based on

FESEM Image	Concentration (%)	Time (min)	Temperature (^{0}C)	Voltage (V)	Average Nanopore Radius (nm)	Average Nanopore Circularity (C)
A	5	5	20	50	7.9	0.73
B	5	9	20	50	8.9	0.61
C	5	20	20	50	9.1	0.57
D	5	30	20	50	11.6	0.56
E	4.7	8	5	35	8.0	0.68
F	4.7	8	5	40	9.6	0.78
G	4.7	8	5	45	13.2	0.66
H	4	20	20	50	12.7	0.55
I	5	20	25	50	14.5	0.67

The aluminium oxide nanopores, categorised based on the geometric feature values of nanopore circularity extracted from the investigated samples are given in the Table 2.

Table 2. The aluminium oxide nanopores are categorised based on the geometric feature values of nanopore circularity extracted from the investigated samples

FESEM Images	Number of Nanopores	Nanopores categorised based on the nanopore circularity (C)		
		C near to 1 (C>=0.8 and C<=1)	C close to 0 (C>=0.3 and C<0.8)	C<0
A	109	46	63	0
B	139	51	88	0
C	35	19	16	0
D	37	11	26	0
E	129	63	66	0
F	221	125	96	0
G	74	30	44	0
H	99	33	66	0
I	86	34	52	0
Total	929	412	517	0

The experimental results are discussed as below:

- As the anodization time is increased (5 mins, 9 mins, 20 mins and 30 mins), keeping constant the concentration (5%), temperature (20^0 C) and voltage (50V);
  - The nanopore radius has gradually increased from 7.9nm to 11.6nm (Fig. 6(1)).
  - The nanopore circularity has decreased from 0.73 to 0.56 (Fig. 7(1)).
- Similarly, as the voltage is increased (35V, 40V, 45V), keeping the concentration (4.7%), time (8 min) and temperature (5^0C) constant;
  - The nanopore radius increased gradually from 8nm to 13.3nm (Fig. 6(2)).
  - The nanopore circularity increased initially from 0.68 to 0.78 when the voltage is increased from 35V to 40V but decreased to 0.66 when the voltage was further increased to 45V (Fig. 7(2)).
- When the concentration (4% and 5%) and temperature (20^0C and 25^0C) was altered keeping time (20 min) and voltage (50 V) constant;
  - The nanopore radius increased from 55.6nm to 67.4nm (Fig. 6(3)).
  - The nanopore circularity increased from 0.55 to 0.67 (Fig. 7(3)).

The extracted feature values of nanopore radius and nanopore circularity are verified by a chemical expert, which proves the exhaustiveness of the proposed results. Further, the total number of nanopores based on the geometric feature values of nanopore circularity which are closer to 1 are 412 nanopores and 517 nanopores are closer to 0, out of total 929 nanopores from all the image data sets (A to I).

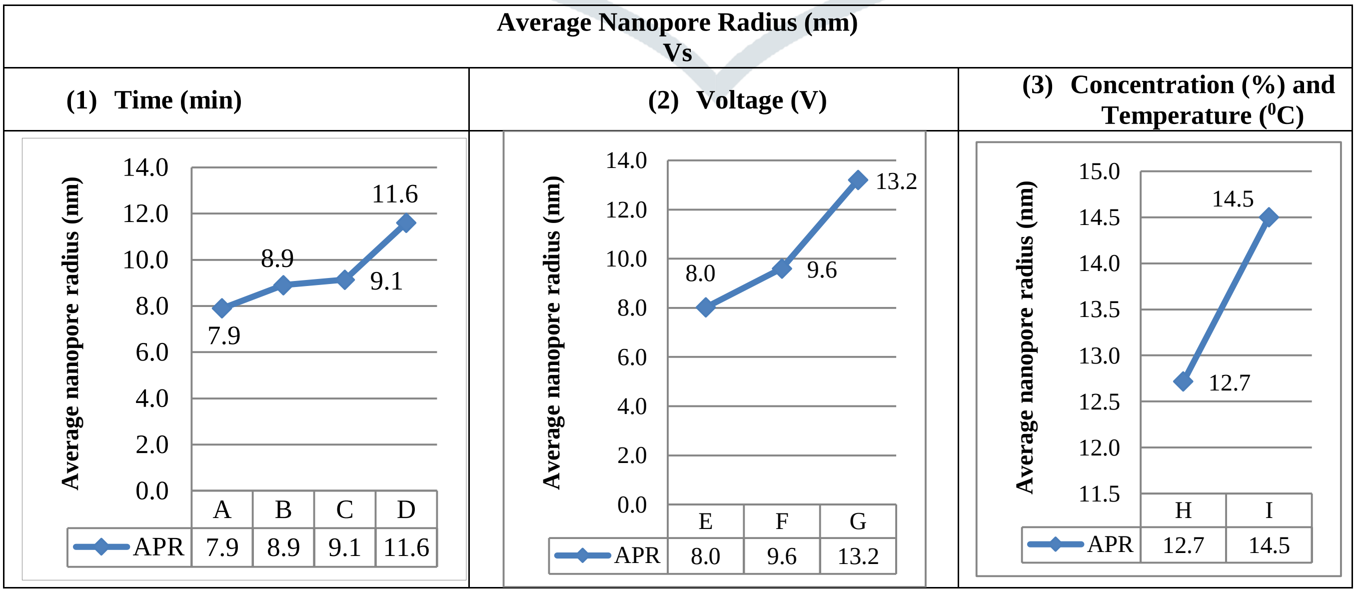
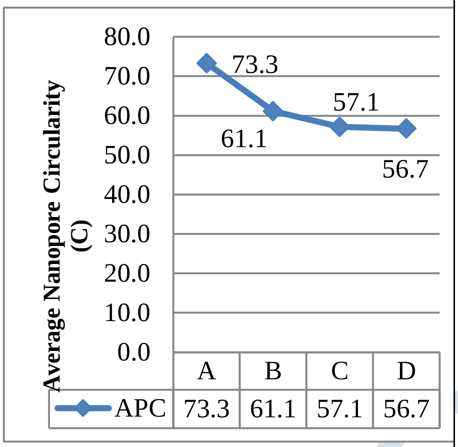
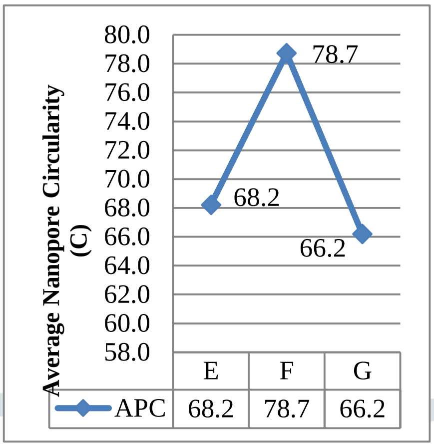


Figure. 6 Nanopore radius in FESEM images with varied anodization time, voltage, temperature and concentration

Average Nanopore Circularity (C) Vs		
(1) Time (min)	(2) Voltage (V)	(3) Concentration (%) and Temperature (^{0}C)





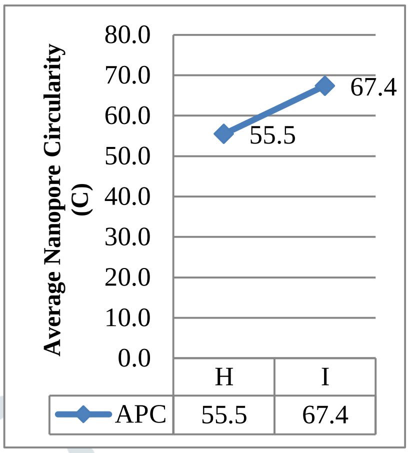


Figure. 7 Nanopore circularity in FESEM images with varied anodization time, voltage, temperature and concentration

V. CONCLUSION

A computerized automated tool is developed to extract and measure geometrical features of nanoporous media. Nanopore radius and nanopore circularity of Anodic Aluminium Oxide (AAO) is determined in the present study on different anodizing parameters. The experimental results illustrate that as the anodization time is increased (5mins, 9mins, 20mins and 30mins)while keeping the constant value in concentration (5%), temperature (20^{0}C) and voltage (50 V), the nanopore radius have gradually increased from 7.9nm to 11.6nm and nanopore circularity has decreased from 0.73 to 0.56. Similarly, as the voltage is increased (35V, 40V, 45V), the nanopore radius increased gradually from 8nm to 13.3nm and the nanopore circularity increased initially from 0.68 to 0.78 and decreased to 0.66 by maintaining the concentration (4.7%), time (8min) and temperature (5^{0}C) constant. When the concentration (4% and 5%) and temperature (20^{0}C and 25^{0}C) has been altered keeping time (20 min) and voltage (50 V) constant, the increase in nanopore radius from 55.6nm to 67.4nm and increase in nanopore circularity from 0.55 to 0.67 is observed. The features drawn from values of nanopore radius and circularity are verified by professional chemical experts, which ensures the exhaustiveness of the results proposed. Further, based on geometric feature values of nanopore circularity, it is observed that, out of 929 nanopores from all the image data sets i.e A to I, 412 nanopores are closer to 1 and 517 nanopores are closer to 0. The measurement of nanopore circularity helps in transport mechanism and predicting flow properties of various porous media relevant to fundamental and industrial applications.

VI. ACKNOWLEDGMENT

The authors are grateful to Indian Institute of Science (IIS), Bengaluru and Department of Chemistry, Rani Channamma University, Belagavi for providing the Al_2O_3 FESEM images and visualizing the manual results.

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