An Improved Method for Measuring the Properties of Aluminium Oxide Nanopore FESEM Images

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Abstract- Characterization of fabricated nano-template plays a vital role in nano engineering and technology. The geometrical properties of the nanopores in synthesized nano membrane depend on the anodizing parameters like concentration of acid, anodizing time, temperature of the bath and voltage applied. The present study exhibits the automated tool to measure the nanoporous media by extracting the geometrical features; nanopore radius and nanopore circularity of anodic aluminium oxide (AAO) by varying anodizing parameters. The experimental results depicts that as the anodization time is increased (5mins, 9mins, 20mins and 30mins), the nanopore radius have gradually increased from 7.9nm to 11.6nm and nanopore circularity has decreased from 0.73 to 0.56 by keeping the constant value in concentration (5%), temperature (20^0C) and voltage (50 V). 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. The increase in nanopore radius from 55.6nm to 67.4nm and increase in nanopore circularity from 0.55 to 0.67 is witnessed 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 extracted feature values of nanopore radius and circularity are verified by a chemical expert, which proves the exhaustiveness of the proposed results.

Keywords- *AAO*, Aluminium Nanopore, Characterization, FESEM, Pore radius, Pore circularity, Nanomaterial, Nanotechnology.

I. INTRODUCTION

Anodic aluminium oxide (AAO)[1] has drawn extensive attention in nanotechnology due to its well-defined pore architecture and suitable corrosion resistance, thermal stability, hardness, abrasion resistance and insulation properties. As a nano-template or host material, AAO plays an essential role for various surface engineering applications, e.g. molecular separation, catalysis, energy storage, electronics, sensors, drug delivery and template synthesis, and it is a component in a diverse range of nanostructured materials in the form of nanodots, nanowires and nanotubes [2-6]. Pore geometry is found useful in describing important flow and transport mechanisms and in predicting flow properties of different porous media relevant to numerous fundamental and industrial applications [10-11]. The benefit of nanotechnology depends on the fact that it is possible to tailor the structures of materials at extremely small scales to achieve specific properties, thus greatly extending the material science toolkit. Using nanotechnology, materials can effectively be made stronger, lighter, more durable, more reactive, more sieve-like, or better electrical conductors, among many other traits. There are many daily used commercial products in the market based on the nanoscale materials and its process.

The importance of nanoscale materials has drawn attention of many nano researchers for the last couple of decades. 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 was proposed by P. Bannigidad and Jalaja U. [13-15].

The objective of the present study is to extract the geometric feature values of nanopore radius and nanopore circularity from the Al_2O_3 FESEM images using global thresholding method. These values are useful in describing the important flow and transport mechanism and in predicting flow properties of different porous media relevant to numerous fundamental and industrial applications.

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, 20^oC and 25^oC temperature, 20 mins time and 50V voltage.

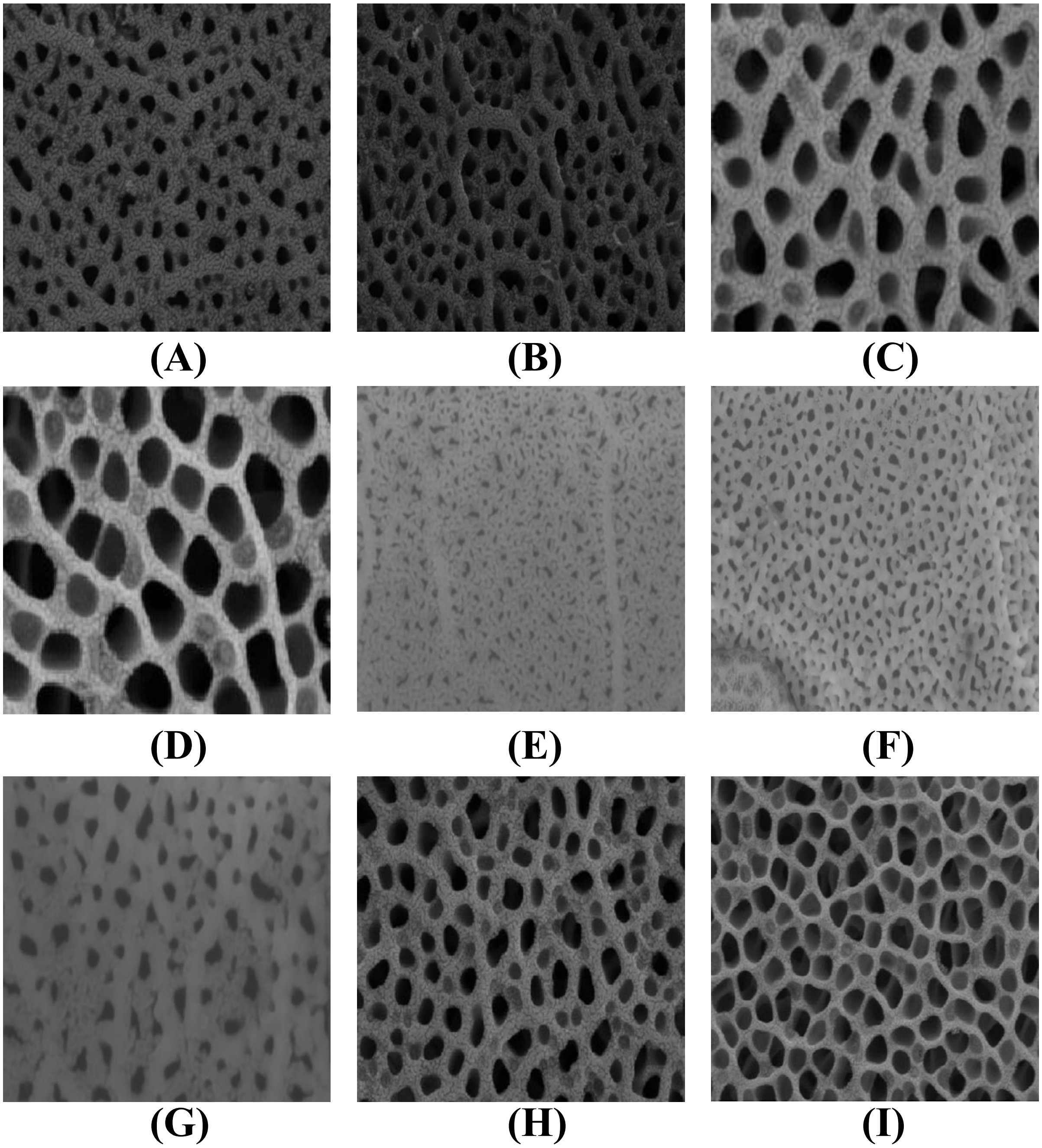

Fig. 1 Top view of aluminium oxide FESEM images

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:

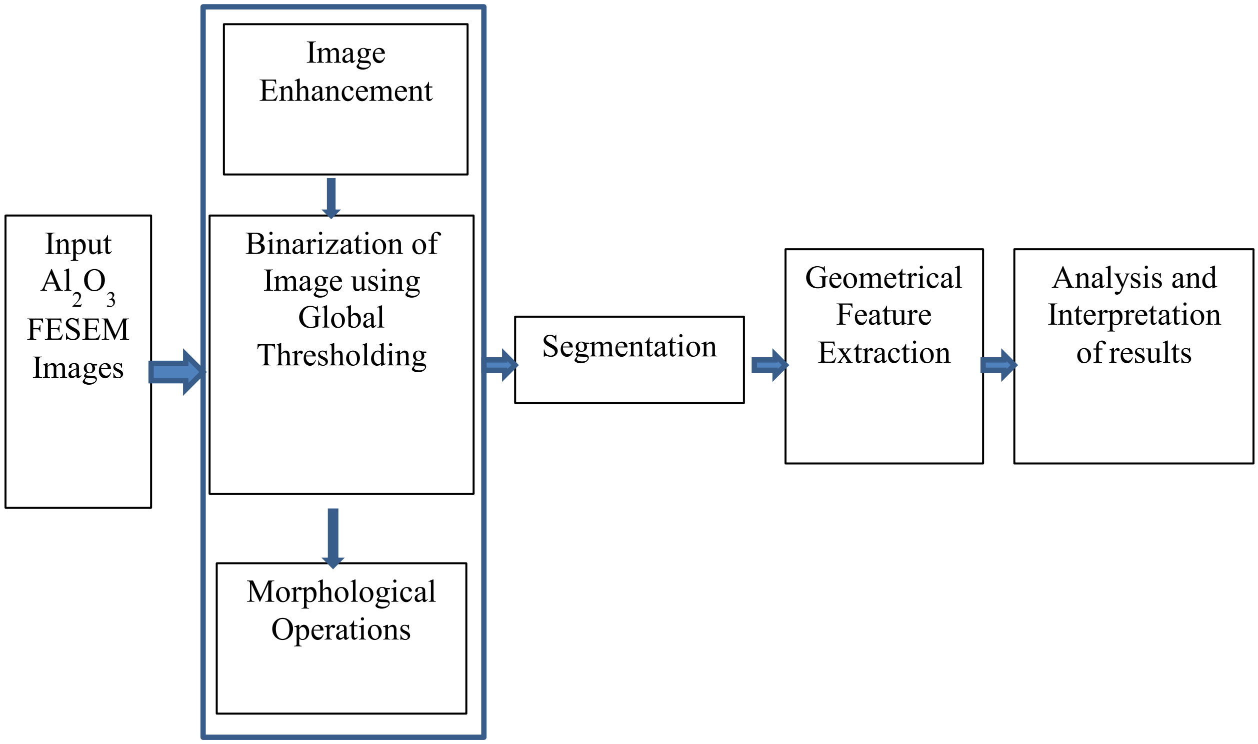

Fig. 2 Flow diagram of proposed method

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).

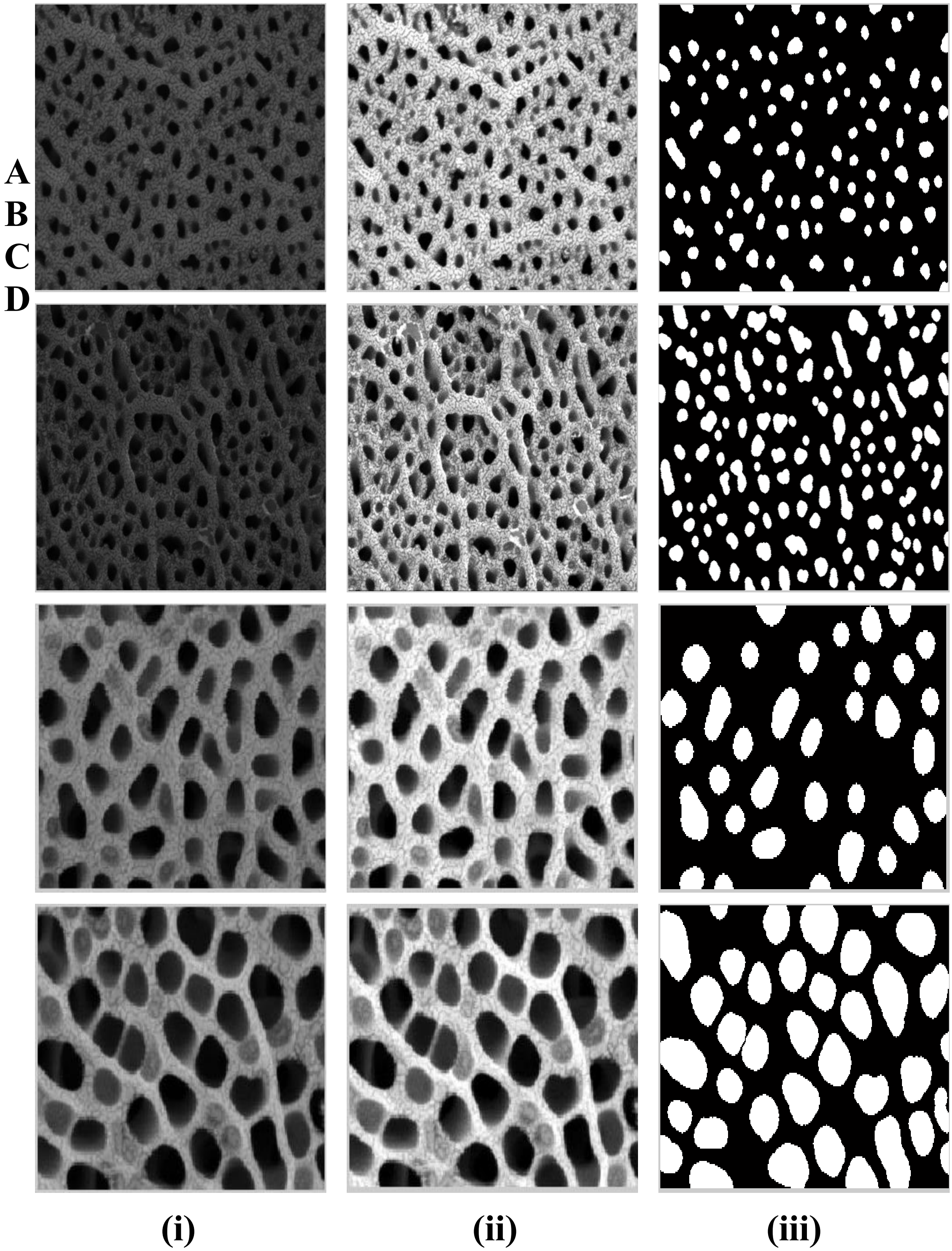


Fig. 3 (i) Original FESEM images captured at different intervals of time, (ii) Grayscale images, (iii) Segmented images

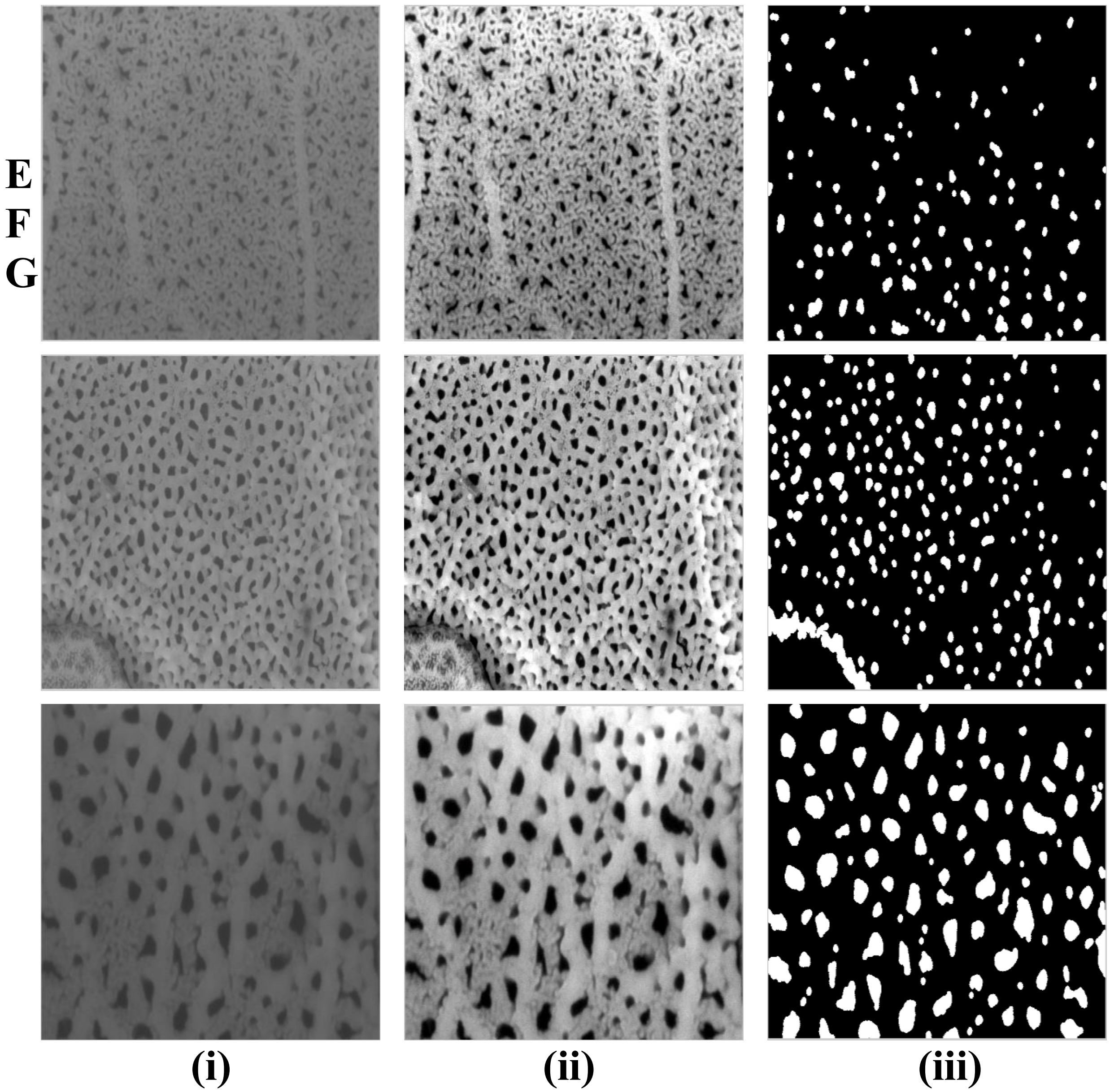


Fig. 4 (i) Original FESEM images captured at different voltage, (ii) Grayscale images, (iii) Segmented images

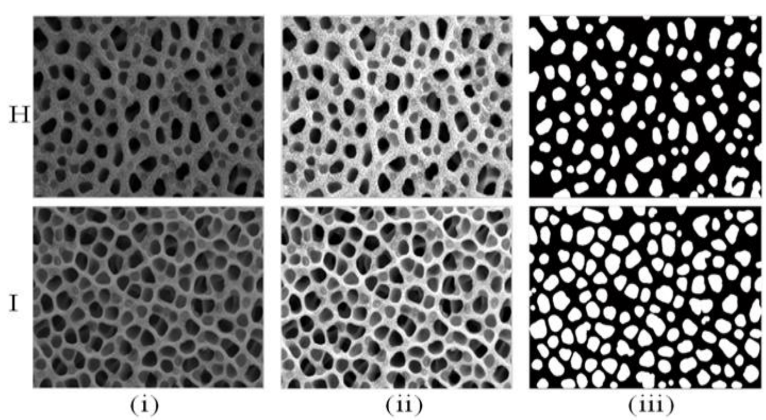


Fig. 5 (i) Original FESEM images captured at different temperature and concentration, (ii) Grayscale images, (iii) Segmented images

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 circularity

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

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 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

- 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).

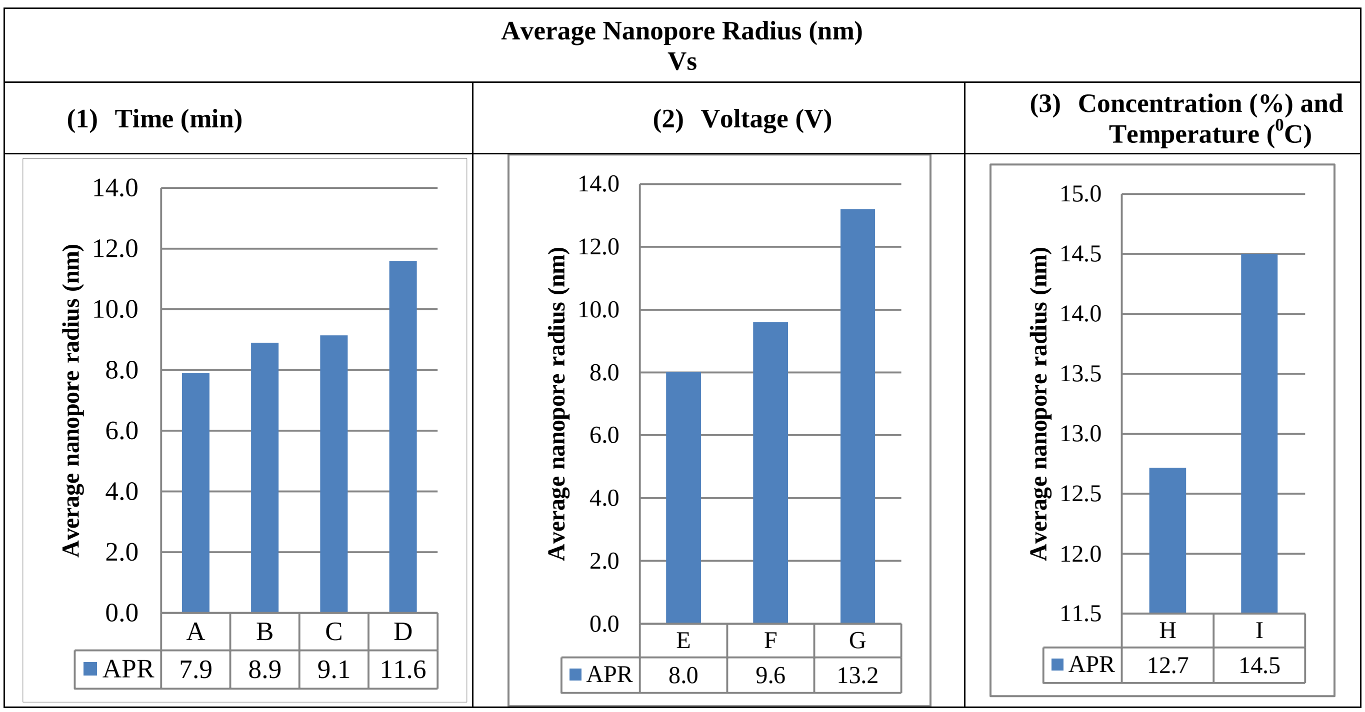
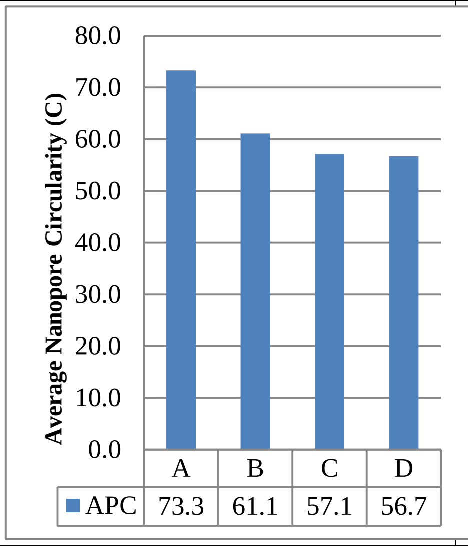
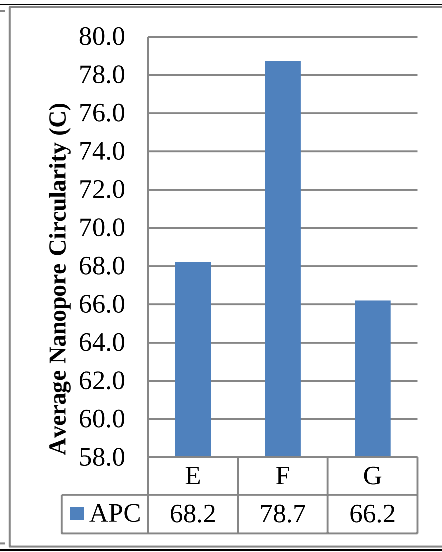


Fig. 6 Nanopore radius in FESEM images with varied anodization time, voltage, temperature and concentration

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





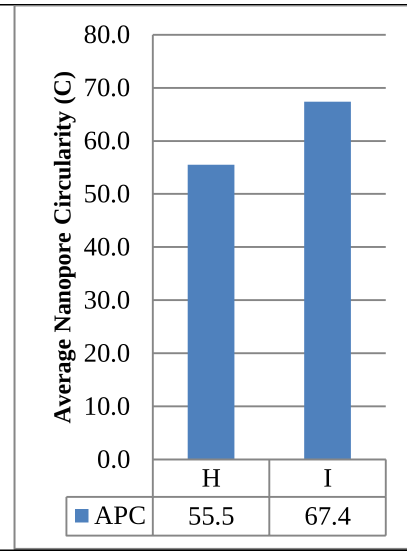


Fig. 7 Nanopore circularity in FESEM images with varied anodization time, voltage, temperature and concentration

V. Conclusion

An automated tool is developed to measure the geometrical features; nanopore radius and nanopore circularity of AAO with the varying anodising parameters. The experimental results depicts that as the anodization time is increased (5mins, 9mins, 20mins and 30mins), and the nanopore radius have gradually increased from 7.9nm to 11.6nm and nanopore circularity has decreased from 0.73 to 0.56 by keeping the constant value in concentration (5%), temperature (20^{0}C) and voltage (50 V). 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. The increase in nanopore radius from 55.6nm to 67.4nm and increased in nanopore circularity from 0.55 to 0.67 was witnessed when the concentration (4% and 5%) and temperature (20^{0}C and 25^{0}C) was altered keeping time (20 min) and voltage (50 V) constant. 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, 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). The extracted properties of aluminium nanoporous images are may be useful in describing the important flow and transport mechanism and in predicting flow properties of different porous media relevant to numerous fundamental and industrial applications.

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