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ORIGINAL ARTICLE

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Synthesis of meso-macro alumina using yeast cells as a bio-template and optimization using a genetic algorithm

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Abstract

Meso-Macro porous alumina was fabricated using yeast cells as a pore-forming agent. Alumina powder synthesis was achieved by a low cost process (recrystallisation of alum).The effect of the pore forming agent on the true porosity, bulk density and thermal conductivity of porous alumina was characterized. The results show that the true porosity increased with the increasing addition of yeast cells. The bulk density and thermal conductivity at room temperature decreased with the increasing yeast addition. A genetic algorithm method was used to minimize the thermal conductivity of the macro-porous alumina based on the amount of yeast cells used, the sintering temperature, and the hold time. The genetic algorithm found that the best thermal conductivity achievable was equal to 0.152 Watt/m. °C at 20wt% concentration of yeast, a sintering temperature of 1230°C and 1.5 hours of soaking time. The experimental value was 0.14 Watt/m. °C and the slight variance between these values were postulated to be due to experimental error in the measurements.

KEYWORDS

alumina, genetic algorithm, pore forming agent, recrystallisation of alum, thermal conductivity, yeast cell

1 | INTRODUCTION

Porous alumina attracts fundamental interest as a model for insulating materials and is an outstanding filtration medium. This is because of its thermal stability, low thermal conductivity, low gas absorption, low thermal capacity, good thermal cycling and shock resistance.[1] Insulating materials display the property of a substance to prevent heat exchange. Commonly, they are used in industrialized furnace lining material, cold containers, stoves, and also a number of applications of aerospace technology.[2–4] Insulating—materials are generally porous, light-weight, with low thermal conductivity, Porous alumina is one of the most the important insulating materials.[5] Many alternative methods have been reported to synthesize porous alumina. These methods include; the partial sintering method,[6] gel casting,[7] freeze casting,[8] the organic foam method,[9] and pore-forming agent method.[10] Among these methods, that using pore-forming agents is the most commonly used and efficacious method.[11]

Pore forming agents can be used as additives for the pore-forming materials in powders (dry pressing) or as a slurry with pore-forming agents (slip casting).[12–14] Many pore-forming agents such as; starch, rice husk, sawdust, organic particulates, graphite and yeast cells are in common use.[11] Yeast cells were used in the synthesis of an alumina with combined macro-/meso porosity. This is because the solid cell wall structure of yeast has a hardness that is capable of supporting stresses during processing and maintaining void space after burnout. Therefore, it was interesting to see if it could withstand the pretreatment procedure.[10]

There are several methods that can be used for optimization such as Fuzzy logic, Taguchi optimization, Antcolony optimization, Hill climbing algorithm, Genetic Algorithm,

etc.[15] Very few optimization techniques use evolving algorithms that provide optimal solutions within a range of data. The Genetic algorithm (GA) is a model of machine learning based on the mechanism of natural selection and natural genetics. This is done by creation of a population of individuals, represented by chromosome.[16]

GA requires an optimization function that represents the empirical data in the form of a relation between the input parameters and the responses.[16] GA are based on the survival of the fittest principles in the nature.[16] Also, the genetic algorithm differs substantially from more traditional search and optimization methods because the genetic algorithm search a population of points in the parallel, not a single point. Also, the genetic algorithm do not require derivative information or other auxiliary knowledge, only the objective function and corresponding fitness levels influence the directions of the search. Additionally, the genetic algorithm use probabilistic transition rules, not deterministic ones.[17] Ultimately, the GA requires three steps to use it; the first step of genetic algorithm is creation of initial population with individuals, where individuals represents a possible solution or variables of the problem, the second step is evaluation of individuals via use of fitness function. Finally, the population is operated by three genetic algorithm operators (reproduction, crossover, and mutation) to produce new population of the points.[17]

In our current work, a GA was utilized to obtain the values of the processing variables that provided a meso-macro porous alumina ceramic with the best possible true porosity, bulk density and thermal conductivity. This work reports the results that were obtained by experimental work which included the preparation of five batches of alumina powders with additives. All these batches were prepared by a slip casting method to achieve the porous alumina bodies. The samples were prepared by using various ratios of yeast cells. Ultimately, the samples were sintered at different temperatures, with various soaking times. The goal of optimization utilizing a GA technique was to be able to reinforce the experiment by optimized values of the true porosity, bulk density and thermal conductivity.

2 | EXPERIMENTAL PROCEDURE

2.1 | Alumina powder preparation

Alumina powder was prepared by the recrystallisation of alum. Briefly, a solution of alum was prepared by adding 78 g of alum to 100 mL of distilled water. Alum was dissolved by using a magnetic stirrer with a speed of 1200 rpm at 100°C and treated with sonication for 3 hours, then aged for 3 hours at ambient temperature. The resulting white precipitate looked like whiskers and was filtered and washed with deionized water several times. After a few hours of the drying at ambient temperature in air, the powder was dried in an oven at 150°C. The dried powder was heat-treated at 1200°C for 2 hours at a heating rate of 5°C/min to obtain α-alumina. The sintered powder was ground in a ball mill before characterization.

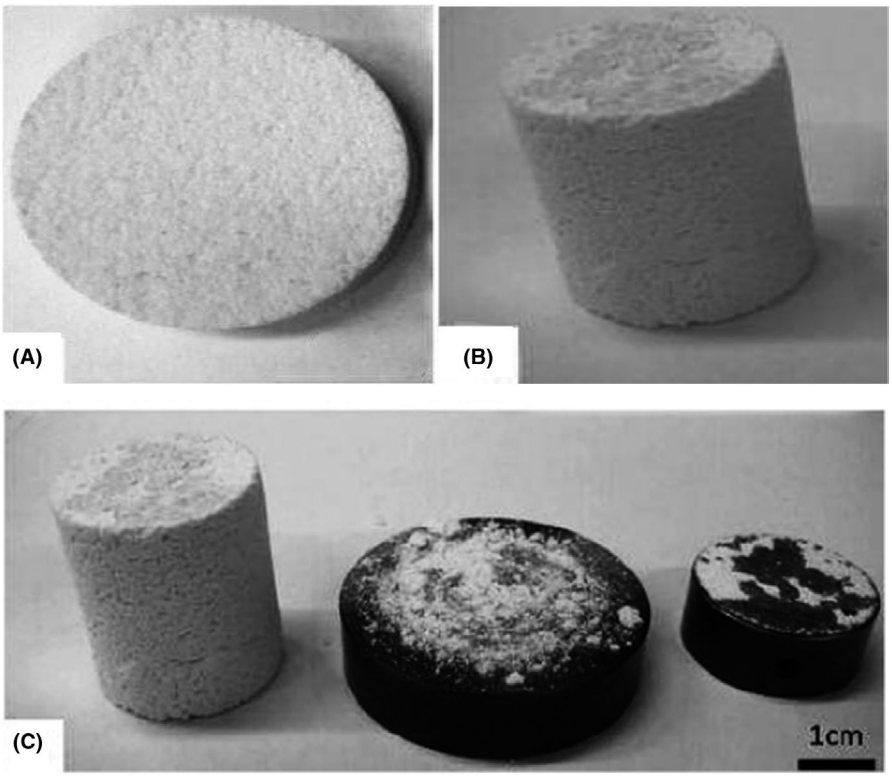

FIGURE 1 Images of the sintered samples

TABLE 1 The processing parameters and results

Yeast %Wt.	Temperature, °C	Socking time (h)	True porosity (%)	Bulk density, g/cm^3	Thermal conductivity (Watt/m. °C)
Batch 1 (0%)	1200	1.5	42.58	1.825	0.7
	1200	3	39.22	1.941	0.77
	1300	1.5	31.65	2.102	0.81
	1300	3	28.28	2.315	1.13
	1400	3	22.39	2.656	1.27
	1500	1.5	16.44	3.05	2.04
	1500	3	14.26	3.6	4.19
Batch 2 (5%)	1200	1.5	69.14	1.212	0.397
	1200	3	67.07	1.268	0.4218
	1300	1.5	67.55	1.196	0.4002
	1300	3	65.31	1.157	0.438
	1400	1.5	67.1	1.064	0.453
	1400	3	62.57	1.122	0.554
	1400	2	62.84	1.078	0.481
	1500	1.5	62.17	1.307	0.521
	1500	3	58.44	1.565	0.68
	1500	2	61.37	1.333	0.537
	1300	2	66.28	1.203	0.433
	1200	2	69.17	1.217	0.401
Batch 3 (10%)	1200	1.5	73.11	1	0.302
	1200	2.5	69.37	1	0.363
	1200	3	69.16	1.238	0.4109
	1300	1.5	71.07	1.074	0.312
	1300	2.5	69.053	1.117	0.376
	1400	1.5	66.43	1.355	0.488
	1500	1.5	69.65	1.166	0.422
	1500	2	66.29	1.235	0.521
	1500	3	63.63	1.278	0.5226
	1400	3	70.77	1.137	0.418
Batch 4 (15%)	1200	2	75.05	1.035	0.252
	1200	3	69.88	1.075	0.3511
	1300	2.5	69.33	1.187	0.371
	1400	1.5	70.27	1.034	0.3488
	1400	2	66.42	1.263	0.4507
	1300	1.5	69.22	1.133	0.389
	1500	1.5	67.42	1.28	0.4211
	1400	3	71.15	1.2	0.3274
	1500	3	66.22	1.357	0.4424
Batch 5 (20%)	1200	1.5	84.11	0.75	0.14
	1200	2	81.97	0.83	0.196
	1300	1.5	76.21	1.03	0.21
	1300	2.5	67.32	1.254	0.477
	1400	2	71.2	1.211	0.305

(Continues)

TABLE 1 (Continued)

Yeast %Wt.	Temperature, °C	Socking time (h)	True porosity (%)	Bulk density, g/cm^3	Thermal conductivity (Watt/m. °C)
	1400	1.5	74.41	1.222	0.202
	1200	3	71.52	1.197	0.328
	1500	2	66.22	1.227	0.421
	1500	1.5	72.75	1.251	0.305

2.2 | Porous alumina preparation

Typically, porous alumina was prepared from the alumina powder (surface area indent 300 m^2/g) in an aqueous solution of yeast cells (Angel Yeast Co. Ltd.) with sugar. Briefly, the suspension was prepared adding alumina powder to 100 mL of distilled water containing varying amounts of yeast cells, 5%, 10%, 15% and 20% wt. The sugar ratio was 1% wt, then there was an addition of 1% wt. deflocculant (Calgon). The materials were mixed using a magnetic stirrer at room temperature and treated with sonication for 2 hours. The yeast cells act on the sugar, forming carbon dioxide gas bubbles that form a closed porous structure. The water addition used was 50%-60% by volume. The resulting slurry was cast in a plaster of Paris mold. After a few hours of drying at room temperature in air, the samples were removed from the mold and dried in an oven at 60°C to remove the moisture. The green bodies were heat-treated at 650°C for 2 hours at a heating rate of 2°C/min to remove the yeast cells. The sintering was conducted at 1200, 1300, 1400 and 1500°C for 90, 120, 180, and 240 minutes with a heating rate of 5°C/min, Figure 1 explains the sintered samples.

2.3 | Characterization of products

The phase of alumina powder was investigated using an *x*-ray diffractometer (XRD 6000, Shimadzu, Japan) in the laboratories of Department of Ceramics Engineering and Building Materials/College of Materials Engineering/ University of Babylon), at room temperature using Cukα radiation (λ = 1.5405 Å), with a scanning speed of 5º/ min from 20° to 70° of 2Θ (Bragg angle) and an applied power of 40 kv/30 mA. The elemental composition was confirmed using Energy Dispersive Spectroscopy (EDS) with an SEM (Bruker XFlash 630 EDS). The sample morphology including the size and shape was examined by scanning electron microscopy (SEM). The bulk density of the sintered samples were measured by using the Archimedes method and true porosity using pycnometer methods. The pore size distribution, specific surface area and the isotherm type were measured using a Brunauer-Emmett-Teller (BET) method which was available in the University Technology Malaysia in Johor Bahru/Malaysia for meso -macro porous structures in the range (20 Å to below ~1500 Å).

2.4 | Design of experimental

Five batches with different concentrations of alumina powder and yeast cells were prepared in order to be able to prepare porous alumina bodies. The experimental population consisted of 47 samples. Each sample was regarded as a chromosome with each chromosome consisting of several genes. These genes were characterized by the input parameters: concentration of yeast, $x(1)$; sintering temperature, $x(2)$; and soaking time, $x(3)$. In the current research, three input parameters with four levels were utilized as shown in Table 1. The fitness function or regression equation was created using a Minitab 17 software program. The regression equation obtained for true porosity can be expressed as follows:

$$\begin{aligned} y(1) = {} & 178 + 4.34 * x(1) - 0.157 * x(2) \\ & - 3.3 * x(3) - 0.2029 * x(1) * x(1) \\ & + 0.000036 * x(2) * x(2) - 1.45 * x(3) * x(3) \\ & + 0.00135 * x(1) * x(2) - 0.150 * x(1) * x(3) \\ & + 0.0059 * x(2) * x(3) \end{aligned} \quad (1)$$

For the bulk density (y_2), the regression equation is;

$$\begin{aligned} y(2) = {} & -2.15 + 0.155x(1) \\ & + 0.00253x(2) + 0.35x(3) - 0.000121x(1) * x(2) \\ & - 0.0186x(1) * x(3) - 0.000061x(2) * x(3) \end{aligned} \quad (2)$$

For the thermal conductivity (y_3), the regression equation is;

$$\begin{aligned} y(3) = {} & -3.47 + 0.368\,x(1) + 0.00273\,x(2) \\ & - 0.47\,x(3) - 0.000263\,x(1) * x(2) \\ & - 0.0277\,x(1) * x(3) + 0.000627\,x(2) * x(3) \end{aligned} \quad (3)$$

where, $y(1)$: True porosity, $y(2)$ bulk density, $y(3)$: thermal conductivity, $x(1)$ concentration of yeast, $x(2)$: sintering temperature and $x(3)$: soaking time. Table 1 illustrates The processing parameters and results.

Based on Table 1 the main effects plots are shown in Figure 2. They show the variation of individual response with the three parameters, i.e. concentration of yeast, sintering temperature and soaking time. In the plots the *x*-axes represents the value of each process parameter and *y*-axes are response value. The horizontal line indicates the mean of the response.

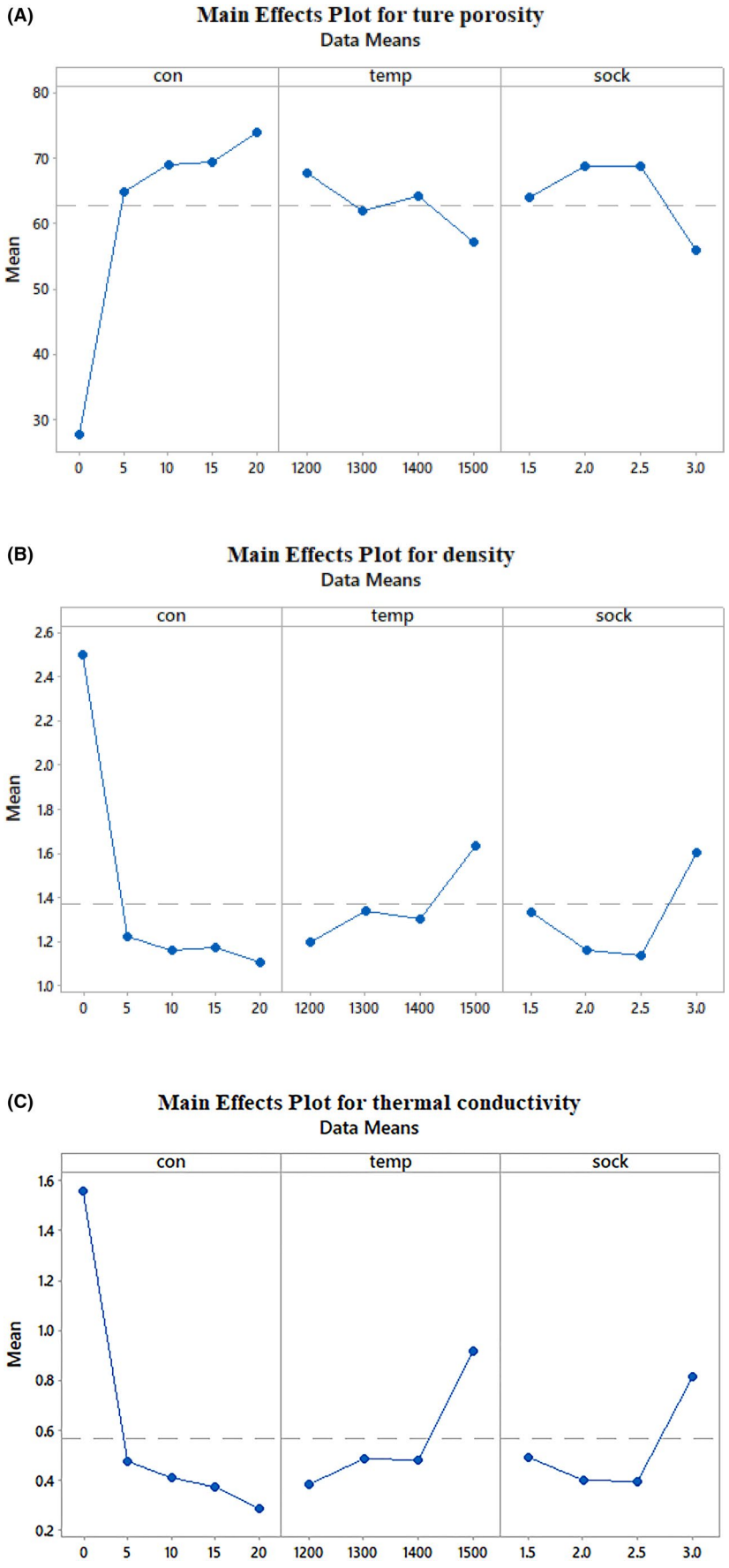

FIGURE 2 Main effects plot for (A) true porosity, (B) bulk density, (C) thermal conductivity

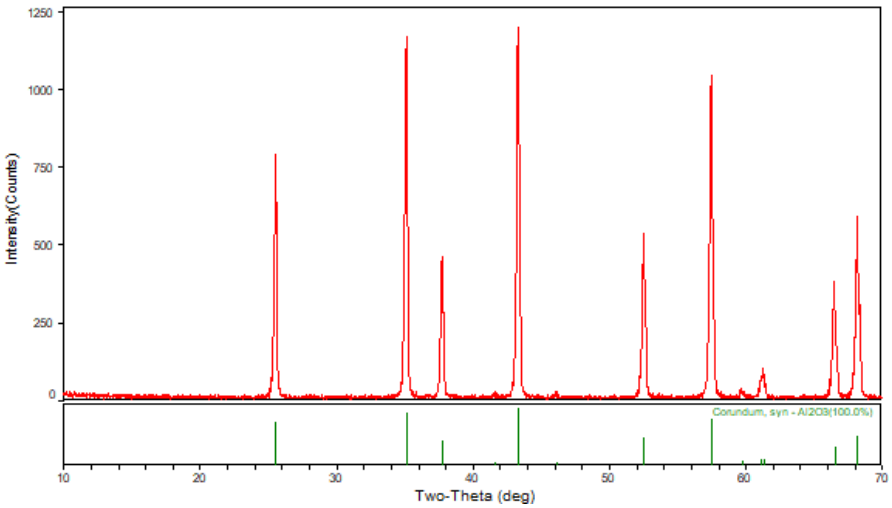

FIGURE 3 XRD pattern of alumina powder calcined at 1200°C for 2 hours with a heating rate of 5°C/min

The main effects plot are used to examine differences between level means for one or more factors based on values from Table 1.

3 | RESULTS AND DISCUSSION

Figure 3 shows the result of XRD analysis of an alumina powder calcined at 1200°C with a heating rate of 5°C/min and soaking time of 2 hours. The very narrow peaks observed indicate that the crystallinity of the alumina powder was very high. The intensity of peaks increased with increased temperature, and the stable phase (corundum) appeared at a sintering temperature greater than 1200°C. The relative intensities obtained from this pattern are in good agreement with (JCPDS) Card No. (00-099-0036) for the hexagonal structure of alumina.

Figure 4 shown the EDS results for the alumina powder prepared at 1200°C for 2 hours. The peaks for Al and O in the EDS spectrum were distinct and their abundance indicates that the formula of the alumina prepared was Al_2O_3.

Figure 5 shows the SEM results of a porous alumina formed with 20%wt. of yeast and sintered at 1200 and 1500°C for 2 hours at a rate of 2°C/min. The SEM shows that the target porous microstructure was achieved. It was a type of meso and macro porous structure with a pore size varying from a few nanometers to larger than 150 nm. The degree of porosity became higher as the yeast content increased, due to the reaction between the yeast cells and sugar leading to the

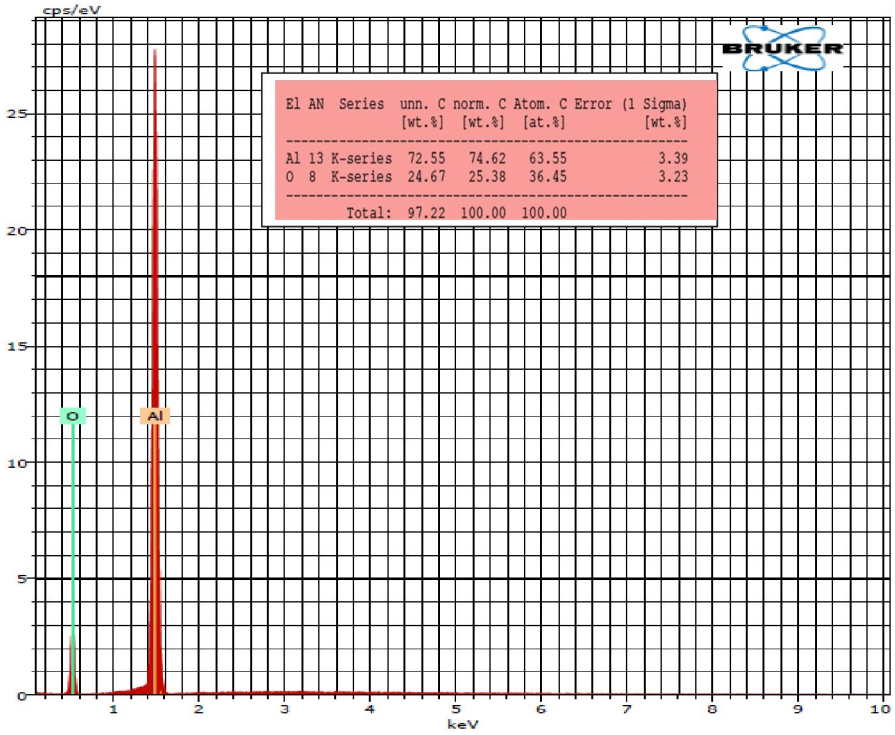

FIGURE 4 EDS analysis of alumina powder

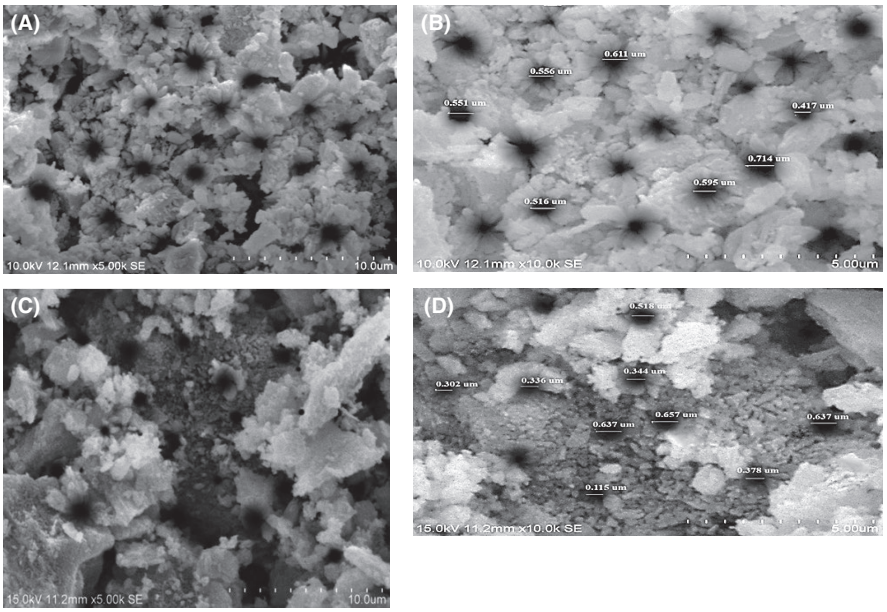

FIGURE 5 SEM of (A)sample with 20wt% and sintering at 1200°C at 5.0KX, (B) sample with 20wt% and sintering at 1200°C at 10.0KX, (C) sample with 20wt% and sintering at 1500°C at 5.0KX, (D) sample with 20wt% and sintering at 1500°C at 10.0KX

formation of carbon dioxide gas bubbles which formed closed pores. The role of bubbles in carbon dioxide is very important in this study as the thermal conductivity of porous material depends on the percentage and quantity of bubbles generated by the yeast reaction with sugar, which contributes to the generation of closed pores because of its insufficient energy to rise to the top of the surface and the forms of open pores.

The presence of open pores is controlled by of the amount of yeast that is not used and which burns during the process of sintering. The size and quantity of pores varies from one sample to another because of changing conditions. The composition in terms of the yeast ratio, as well as the temperature of the sintering and the period of stay, are all reasons to change the size and quantity of the bubbles.

Usually, some volume percentage of large spherical pores can exist in the initial pure Al2O3 green bodies, because of the nonuniform particle arrangement. From the viewpoint of mechanical stability, the spherical pores are more stable than the large pores of other shapes. The unique pore structures in the alumina bodies' specimens were further confirmed by the change in their peak pore size, which represents the diameter of the pore channels between the large solid spheres. Figure 6 shows that the peak pore size of the alumina bodies' specimens increased during densification. However, the peak pore size of the pure Al_2O_3 sintered specimens decreased as the densification progressed.

At the higher sintering temperature the dense microstructure failed to form. This is thought to be because, high temperatures promote the increased grain growth and decomposition of the alumina powder. It is noted that the processing of alumina samples at high temperatures (higher than 1400-1500°C) leads to exaggerated grain growth and decomposition. While, large pores were formed into bodies sintered at 1200°C.

Figure 6A1, shows the N_2 adsorption/desorption isotherms for the samples synthesized at different sintering temperatures. Figure 6A2, shows the samples with a type III isotherm with a hysteresis loop located at the relative pressure (P/P_o) range of 0.15-1, with an average pore size of 90.2554 nm. This indicates a macro-porous structure as shown in Table 2. Figure 6B1, shows samples with a type II isotherm with a hysteresis loop at relative pressure (P/P_0) range of 0.01-1, with average pore size (4.3085 nm) which again indicates a meso-porous structure. The increase in sintering temperature leads to a decrease in the pore volume. Moreover, at high sintering temperatures, the particles became more regular and some inter-particle pores may have disappeared. While, the materials produced at low sintering temperatures showed a high N_2 adsorption indicating a large surface area. The adsorption capacity of the synthesized samples decreases with an increase in sintering temperature.

3.1 | Genetic algorithm

The initial step of a GA is to design the first population with individuals, where the individuals characterize a practical solution or chromosomes. The population consists of the individuals or the chromosomes which forms the mating pool. The chromosome is represented by the parameters of the problem. In this case the concentration of yeast, $x(1)$; sintering temperature, $x(2)$; and soaking time, $x(3)$. Represent the parameters or the variables. The population type is a bit string and their size is 56 individuals. As the number of the generation increases the average fitness value of the entire generation increases, and the individuals in the population get closer to the optimum point. The second step is an analysis of the individuals through a fitness function. The population was managed through using three GA

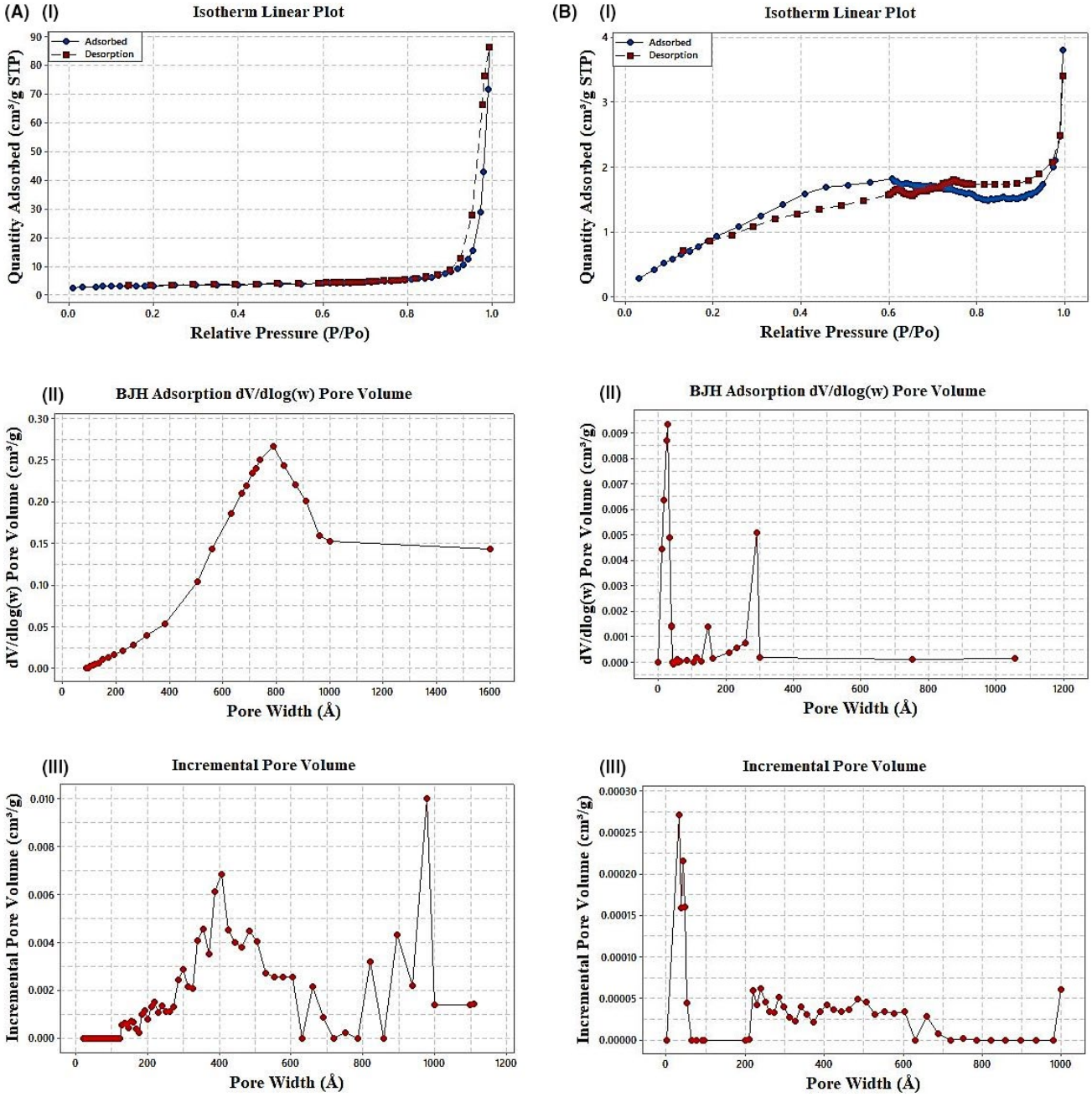

FIGURE 6 BET result of porous alumina (A1, A2, A3) of sample with 20wt% and sintering at 1200°C, (B1, B2, B3) of sample with 20wt% and sintering at 1500°C

managers (crossover, reproduction, and mutation) to create the new population of points. The crossover exchanges the genetic material of the two individuals to create two new individuals. While the mutation process changes the genetic material of an individual. The genetic operators are applied to the individuals of the population until an optimum solution of the problem is found.[12] The GA option as a tool for optimization within the Matlab software program was utilized to characterize solutions of the optimization problems. Each property (porosity, bulk density, and thermal conductivity) was optimized separately Table 3.

3.2 | The output solution

The objective value result in the Matlab software program was at a maximum true porosity 84.37%, minimum bulk density 0.792 g/cm^3 and the minimum of the thermal conductivity 0.152 Watt/m. °C. Figure 7A illustrated the fitness value versus generation, and at the same time Figure 7B exhibits the best individuals or chromosomes at this value of true porosity. This outcome is in reasonable agreement with the experimental value 84.11%. Figure 8A demonstrated the fitness value versus generation, and (Figure 8B) shows the best individuals

TABLE 2 The summaries of BET result for prepared samples

Sintering temperature	BET result					
	BET Surface area (m^2/g)	Langmuir Surface area (m^2/g)	t-Plot external surface area (m^2/g)	BJH Adsorption average pore width (A)	BJH Desorption average pore width (A)	Isotherm type
1200°C	85.22	87.133	90.211	902.554	452.081	Type III
1500°C	7.316	13.416	13.402	43.085	46.436	Type II

TABLE 3 The optimum input and output parameters

Properties	Optimum parameter			Experimental value	Prediction value via GA	Relative error
	Con.of yeast	Temp. sintering	Soak. Time			
True porosity	20	1210	1.5	84.11	84.372	0.00311
Bulk density	20	1380	3	0.75	0.794	0.058
Thermal conductivity	19.5	1230	1.5	0.14	0.152	0.085

or chromosomes at this value of minimum bulk density. Figure 9A illustrated the fitness value versus generation, and at the same time. Figure 9B exhibits the best individuals or chromosomes at this value of thermal conductivity. This outcome is again in reasonable agreement with the experimental value 0.152 Watt/m. °C.

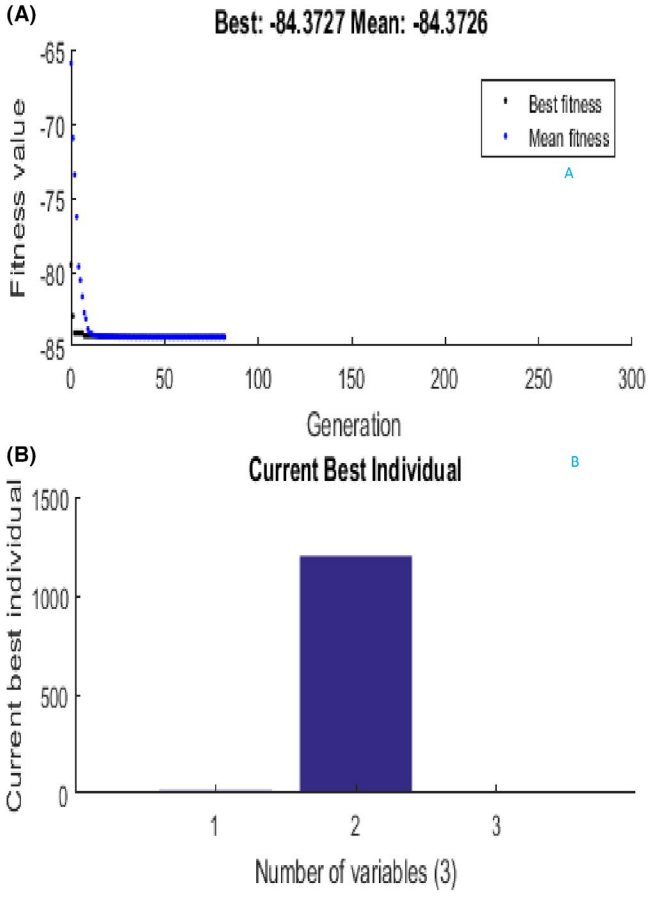

FIGURE 7 A, The generation versus the fitness value. B, The best individual for true porosity

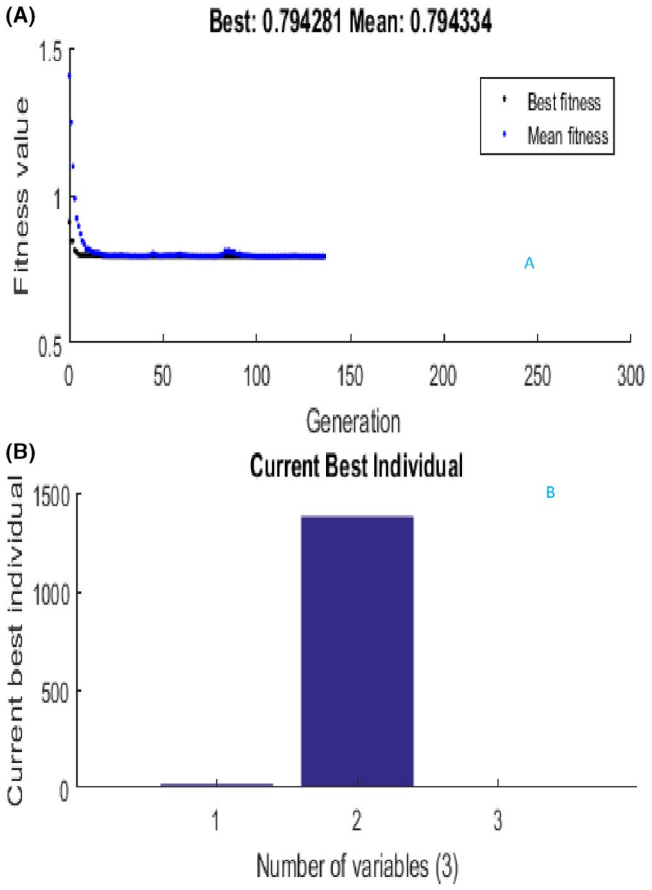

FIGURE 8 A, The generation versus the fitness value. B, The best individual for bulk density

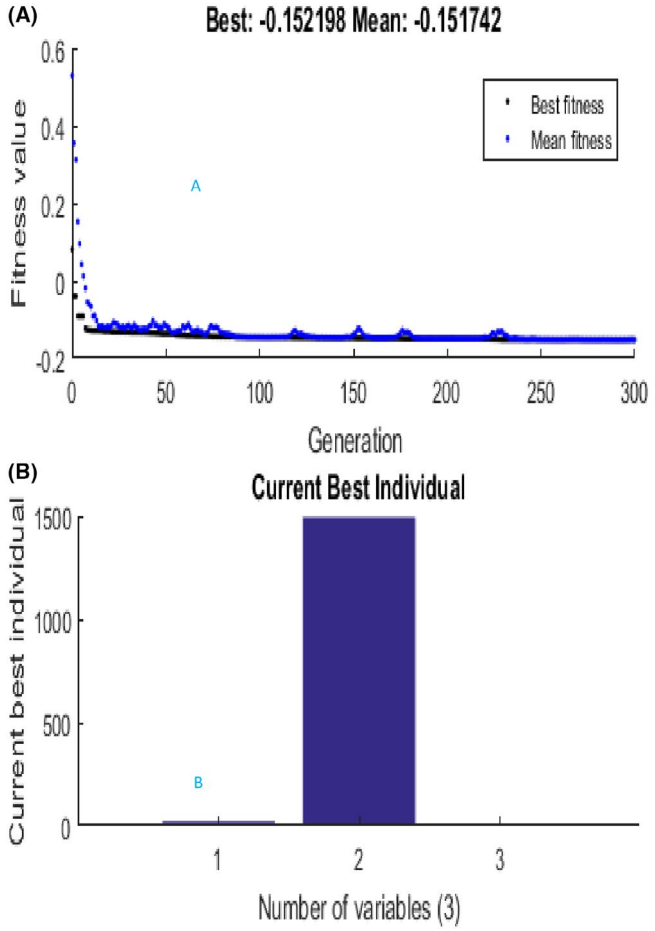

FIGURE 9 A, The generation versus the fitness value. B, The best individual for thermal conductivity

Factors such as moisture, the presence of agglomerates, and other variables that weren't controlled were likely to have contributed to the differences between predicted and experimentally measured values.

4 | CONCLUSIONS

This study showed that a high purity corundum alumina powder with a Hexagonal structure can be successfully prepared by using the recrystallization of alum method. Macro porous alumina was successfully prepared via using a slip casting method, additionally utilizing yeast cells as a pore forming agent with small amount of sugar to produce carbon dioxide gas bubbles that formed closed pores. The porosity obtained was in the range 14.26%-84.11% and thermal conductivity decreased from (4.19-0.14 Watt/m. °C). The increase in the porosity and a decrease in thermal conductivity increased as the percentage ratio of yeast cells increased and at a lower sintering temperature. The GA was used to optimize the problem parameters. The best thermal conductivity was (0.152 Watt/m. °C) at 20wt% concentration of yeast, sintering temperature at 1230°C and 1.5 hours of soaking time. The corresponding experimental value was (0.14 Watt/m. °C). The slight difference between these values, it was probably attributable to experimental error in measurements.

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CONFLICT OF INTEREST

This study was funded by ourselves without support from any company. Conflict of Interest: Authors didn't receive research grants from any Company. As well as, the authors didn't receive a speaker honorarium from any Company. Furthermore, the authors declare that they have no conflict of interest.

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SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of the article.

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