UNIVERSITI PUTRA MALAYSIA

EXPERIMENTAL AND NUMERICAL INVESTIGATION OF PLAIN AND NI-REINFORCED POROUS ALUMINA CERAMICS COMPOSITES PRODUCED WITH AGRO-WASTE PORE FORMERS

DELE-AFOLABI TEMITOPE THEOPHILUS

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By

DELE-AFOLABI TEMITOPE THEOPHILUS

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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Chairperson: Azmah Hanim Mohamed Ariff, PhD
Faculty: Engineering

The mechanical and corrosion resistance properties of porous alumina ceramics are of utmost importance in understanding their operational behavior if they are to stand the test of time. Recently, porous alumina systems have been considered suitable for application in wide-ranging industrial processes that require extreme service conditions such as high temperatures and corrosive mediums due to their satisfactory thermal, mechanical and corrosion resistance properties. However, due to the inherent brittleness of ceramics and their high sensitivity to thermo-mechanical loading, large-scale production of porous alumina components for the above applications is constrained. In the present study, the singular effect of different pore formers (rice husk and sugarcane bagasse) as well as the joint effect of these pore formers and nickel (Ni) reinforcement on the mechanical and corrosion resistance of plain and Ni-reinforced porous alumina ceramics composites have been studied respectively. Experimental results showed that the mechanical properties of the plain porous alumina ceramics decreased with rising pore former content (hardness, tensile stress and compressive stress of 529.1-26HV, 20.4-1.5MPa and 179.5-10.9MPa respectively). Moreover, higher mechanical properties were observed in the SCB-graded samples up to the 15wt% PFA mark, while beyond this point, the silica peak present in the RH-graded samples favored their relatively higher value. The corrosion resistance evaluation of the plain porous alumina ceramics showed that the RH and SCB graded samples demonstrated superior corrosion resistance in strong acid and strong alkali mediums respectively. For the Ni-reinforced porous alumina composites, an inverse relationship was established between the mechanical properties and Ni reinforcement. Overall, maximum hardness, tensile stress and compressive stress values of 167.3HV, 12.6MPa and 55.3MPa respectively were exhibited by the RH-graded porous alumina composite reinforced with 2wt% Ni. Relative to the plain porous alumina series, the RH-graded composites exhibited a better corrosion resistance in the corrosive mediums as compared with the SCB-graded counterparts which demonstrated reduced performance in both mediums. Moreover, superior corrosion resistance was observed in the RH-graded porous

alumina composite reinforced with 2wt% Ni. The Levenberg Marquardt Back Propagation Artificial Neural Network (LMBP ANN) was deployed as an artificial intelligence model to characterize the plain and Ni-reinforced porous alumina ceramics composites developed in the present study. The inputs of the models developed include the sample formulation and the corroding time while the outputs are the density, porosity, hardness, compressive stress, tensile stress, tensile modulus, mass loss in NaOH and mass loss in H_2SO_4. The accuracy and performance efficiency of the developed models (ANN I and ANN II) were confirmed by the large coefficient of determinant (≥ 0.95) registered for the plots of all the experimental results against their corresponding LMBP ANN predicted results. A Graphical User Interface was designed to create a user friendly platform that provides users with real time characterization of the plain and Ni-reinforced porous alumina ceramics composites.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan ijazah Doktor Falsafah

PENYELIDIKAN BEREKSPERIMEN DAN BERANGKA KE ATAS SERAMIK ALUMINA LIANG ASLI DAN DIPERKUAT-NI DIHASILKAN MELALUI PEMBENTUK LIANG BAHAN BUANGAN AGRO

Oleh

DELE-AFOLABI TEMITOPE THEOPHILUS

Mei 2018

Pengerusi: Azmah Hanim Mohamed Ariff, PhD
Fakulti: Kejuruteraan

Ciri-ciri mekanikal dan rintangan kakisan seramik alumina berliang adalah sangat penting dalam memahami tingkah laku operasinya jika digunakan menentang ujian masa. Baru-baru ini, sistem alumina berliang telah dianggap sesuai untuk digunakan dalam pelbagai proses perindustrian yang memerlukan keadaan perkhidmatan yang mencabar seperti suhu tinggi dan terdedah kepada medium mengakis kerana ciri-ciri rintangan haba, mekanikal dan kakisan yang memuaskan. Walau bagaimanapun, disebabkan kerapuhan seramik dan sensitiviti yang tinggi kepada pemuatan termo-mekanik, pengeluaran komponen alumina berliang secara meluas untuk aplikasi di atas terkekang. Dalam kajian ini, kesan tunggal pembentuk liang yang berbeza (sekam padi dan hampas tebu) serta kesan bersama pembentuk liang dan nikel (Ni) sebagai tetulang pada sifat mekanikal dan rintangan kakisan komposit seramik alumina liang asli dan diperkuat nickel telah dikaji. Keputusan eksperimen menunjukkan bahawa sifat mekanik seramik alumina berliang asli berkurang dengan kandungan pembentuk liang yang meningkat (kekerasan, tegasan tegangan dan tekanan mampatan 529.1-26HV, 20.4-1.5MPa dan 179.5-10.9MPa masing-masing). Lebih-lebih lagi, sifat mekanik yang lebih tinggi diperhatikan dalam sampel yang mengandungi SCB sehingga nilai PFA% 15wt, melebihi nilai ini, puncak silika yang hadir dalam sampel RH yang dinilai adalah lebih tinggi. Penilaian rintangan kakisan seramik alumina berliang asli menunjukkan bahawa sampel RH dan SCB yang dinilai memberikan rintangan kakisan yang unggul dalam asid dan medium alkali kuat. Untuk komposit alumina berliang yang diperkuat Ni, hubungan songsang telah ditubuhkan di antara sifat-sifat mekanik dan kandungan pengukuh Ni. Secara keseluruhannya, kekerasan maksimum, tegasan tegangan dan nilai tegasan mampatan masing-masing 167.3HV, 12.6MPa dan 55.3MPa dipamerkan oleh komposit aluminium berliang RH yang diperkuat dengan 2wt% Ni. Merujuk kepada sampel alumina berliang asli, komposit RH yang dinilai mempunyai rintangan kakisan yang lebih baik dalam medium hakisan berbanding dengan sampel mengandungi SCB yang menunjukkan penurunan prestasi dalam kedua-dua medium. Selain itu, rintangan kakisan yang lebih tinggi diperhatikan dalam komposit

aluminium berliang RH yang diperkuat dengan 2wt% Ni. Levenberg Marquardt Back Propagation Network Neural Artificial (LMBP ANN) telah digunakan sebagai model kecerdasan buatan untuk mencirikan komposit seramik alumina yang asli dan diperkuat Ni yang dibangunkan dalam kajian ini. Data masuk model-model yang dibangunkan adalah termasuk rumusan sampel dan masa kakisan manakala data keluar adalah ketumpatan, keliangan, kekerasan, tegasan mampatan, tegasan tegangan, modulus tegangan, kehilangan berat dalam NaOH dan kehilangan berat dalam H_2SO_4. Ketepatan dan kecekapan prestasi model yang dibangunkan (ANN I dan ANN II) telah disahkan oleh pekali penentu yang besar (≥ 0.97) yang diperolehi dari mencarta semua keputusan percubaan terhadap keputusan anggaran LMBP ANN. Grafik antara muka untuk pengguna direka bentuk untuk mewujudkan pelantar mesra pengguna yang menyediakan pencirian antara masa yang nyata untuk komposit seramik alumina polos dan tegangan Ni-diperkuat.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

Azmah Hanim Mohamed Ariff, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Norkhairunnisa Mazlan, PhD
Senior Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Shafreeza binti Sobri, PhD
Senior Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Prof Recep Calin, PhD
Professor
Kirikkale University
Turkey
(Member)

ROBIAH BINTI YUNUS, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

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LIST OF SYMBOLS AND ABBREVIATIONS

Al_2O_3	Alumina
ANN	Artificial Neural Network
ASTM	American Society for Testing and Materials
BF	Bright Field
CMC	Ceramic Matrix Composite
DF	Diametral Fracture
DTA	Differential Thermal Analysis
EDS	Energy Dispersive Spectroscopy
FESEM	Field Emission Scanning Electron Microscope
FETEM	Field Emission Transmission Electron Microscope
FIB	Focused Ion Beam
g	Grams
GUI	Graphical User Interface
GUIDE	Graphical User Interface Development Environment
HCl	Hydrochloric Acid
H_2SO_4	Sulphuric Acid
HV	Vickers Hardness
ISO	International Organization for Standardization
Kg	Kilogram
LMBP	Levenberg-Marquardt Back Propagation
LRF	Load Region Fracture
m	Meters
MAE	Mean Absolute Error
MPA	Mega Pascal
MATLAB	Matrix Laboratory
N	Newton
NaOH	Sodium Hydroxide
Ni	Nickel
$NiAl_2O_4$	Nickel Aluminate Spinel
$N_3Al_2SiO_8$	Nickel Alumosilicate Spinelloid
P_c	Critical Porosity Limit
PFA	Pore-forming Agent
RH	Rice Husk
RHA	Rice Husk Ash
RMSE	Root Mean Square Error
rpm	Revolution Per Minute
SAED	Selected Area Electron Diffraction
SCB	Sugarcane Bagasse
SiO_2	Silicon Dioxide / Silica
TCF	Triple Cleft Fracture
TGA	Thermogravimetric Analyzer
UTM	Universal Testing Machine
vol%	Volume Percent
wt%	Weight Percent
XRD	X-ray Diffractometer

XRF	X-ray Fluorescence Spectroscopy
ε_t	Tensile Strain
E	Tensile Modulus
σ_c	Compressive Stress
σ_t	Tensile Stress

CHAPTER 1

INTRODUCTION

1.1 Background of Research

In recent times, the utilization of ceramic materials as household hardware, industrial use and structural applications has received a tremendous acceptance amidst various end users owing to their high thermal stability, corrosion resistance, good wear resistance, poor conductivity, excellent mechanical properties and others. This group of materials has surged the interest of researchers by delving further into advancing the development of ceramic products that can suit other specific requirements.

Thus far, studies have shown the major setback in the use of ceramic materials for structural applications to be the constant evolution of pores within the microstructure which serves as fracture sites thereby deteriorating the structural integrity of this group of materials. However, systematic control of these pores can be channeled towards the development of porous ceramic materials suitable for application in wide-ranging technologies such as filtration, thermal insulation, food processing, biomedical implants and others.

To a large extent, ample homogenous porous ceramics have been largely manufactured through the utilization of state-of-the-art processing methods. One of such processes is the employment of pore-forming agents (PFAs) in both solid and liquid forms. In spite of the multiplicity of processing technologies, the pore-forming agent (PFA) approach has far been preferred over other methods for small-scale fabrication of porous ceramics, owing to its simplicity, economic viability and easy accessibility of materials. Moreover, quite a number of agricultural wastes (lignocellulosic biomass) have shown remarkable potentials in this regard. Therefore, taking into consideration the rise in respiratory health hazards resulting from the incessant burning of fields, the ever stricter environmental policy acts can be complied with by efficiently utilizing agro-waste materials, thereby enhancing adequate health safety and promoting the sustainability of the ecosystem.

More so, with the rising metric tons of lignocellulosic biomass deposit in fields of agriculture dependent countries, studies (Irfan et al., 2014; Ahmed et al., 2015) in recent times have focused on the eradication of the hazardous methods for agricultural waste management and boosting the economic benefit from this group of materials. Hence, it is imminent to tap into this abundantly available agro-waste materials like the rice husk and sugarcane bagasse which can be channeled towards the fabrication of valuable porous ceramics owing to the significant silica content in these waste materials. Similarly, with the groundbreaking advances made thus far in the field of material science, it is important to maximize the use of material properties efficiently, in order to achieve high reliability and maintain an acceptable level of performance efficiency. In recent years, the conventional means of achieving such feat has being through the addition of alloying elements which often brings improvement to the properties of the matrix material. Appropriate selection of the reinforcement material could desirably upgrade the intrinsic properties of porous ceramic systems.

With alumina (Al_2O_3) as the matrix, nickel (Ni) as the reinforcement as well as sugarcane bagasse (SCB) and rice husk (RH) as the pore-forming agents, both plain and Ni-reinforced porous alumina ceramics composites with formulations Al_2O_3-PFA and Al_2O_3-Ni-PFA were developed in the present study. Thereafter, the samples were subjected to series of conventional laboratory testing to obtain the mechanical and corrosion resistance properties.

1.2 Problem Statement

Over the past decades, highly porous ceramic membranes have made rapid progress in broad-based and strategic industrial technologies such as thermal insulation, bone tissue engineering, molten metal filtration, wastewater treatment and others. Meanwhile, the utilization of pore-forming agent processing technique, continues to dominate the manufacturing space in this field of study due to its production sustainability, ease of handling and economic feasibility. However, porous ceramic materials shaped with natural organic matters such as starch have exhibited a constrained pore geometry within a range of <100μm (mean particle sizes of approx. 5, 14 and 50μm for rice, cassava and potato starch particles respectively) (Sandoval et al., 2012; Sandoval et al., 2017). For this reason, it becomes necessary to seek more flexible alternatives under the natural organic PFA category for the development of porous ceramic materials.

Meanwhile, alumina has been the most widely used ceramic material in the fabrication of porous ceramic components due to the exceptional mechanical and corrosion resistance properties demonstrated by this group of ceramics. However, investigations have shown that the inherent brittleness and high sensitivity to post-fabrication processes are obstacles restraining the extensive application of porous alumina ceramics especially in separation membrane units where the infiltration of hot corrosive slurry at marked transmembrane pressure is a major concern (Li et al., 2013; Qin et al., 2015). For these reasons, it is imperative for researchers and industrial experts to explore the composite approach in revamping the traditional porous alumina ceramics so they can thrive well under extreme service conditions.

Recently, nickel (Ni) has been well acknowledged as an excellent reinforcement for suppressing the brittleness of ceramics due to its high tensile strength and toughness, superior corrosion resistance, high melting temperature among others (Lu et al., 2000; Fung and Wang, 2014). The exceptional combination of properties between the alumina matrix and the nickel particulates in the investigations above resulted in the enhancement of mechanical properties and microstructural refinement of the composites relative to the plain counterpart. However, these investigations only focused on fully dense Al_2O_3/Ni composites fabricated either through preform infiltration or hot isostatic pressing. Moreover, drawbacks such as matrix deformation and pore blockage constrain the utilization of the fabrication techniques mentioned above for the development of porous ceramic composites. Meanwhile, most of the published literatures to date have reported the preparation of porous ceramics composites by using multi-phasic ceramics approach (Sun et al., 2017), polymer reinforcements (Fu et al., 2018) and fibre reinforcements (Ritcher and Peters, 2016; Han et al., 2017). Nonetheless, constraints such as difficulty in reinforcement dispersion, impaired mechanical properties and pore size limitation impede the implementation of these conventional routes in the development of porous ceramics composites designed for emerging technologies, where large pores and high mechanical performance are required.

Therefore, in view of achieving the requisite properties needed for porous ceramics to thrive under robust service conditions, the present study presents the utilization of agro-waste pore-forming agents (rice husk and sugarcane bagasse) and nickel reinforcement in developing plain and Ni-reinforced porous alumina ceramics composites; an approach which is lacking in the existing literature. Through the incorporation of rice husk (RH) and sugarcane bagasse (SCB) powders as pore-forming agents as well as nickel (Ni) as the metal phase reinforcement in the alumina (Al_2O_3) matrix, it is expected that the exceptional properties derived from the cluster of these materials will go a long way in eliminating the drawbacks affecting traditional porous ceramic materials.

1.3 Significance of Study

Ongoing comprehensive overview of the impact of greenhouse gas emissions on climate change has heightened the sensitization and interest of researchers from all academic spheres in channeling their resources towards the sustainability of the planet by adopting the “Going green” revolution. With a view to promoting safe management practices, materials experts have successfully recycled and reused agricultural wastes in several production areas. Therefore, unlocking further revenue generation for countries that depends highly on agriculture for revenue generation and also enhances the zero waste concept in particular for developing nations that are yet to comply with safe waste management practices.

Meanwhile, owing to the all year round cultivation, high demand and abundant availability, residues from the monocotyledon group (e.g. rice, wheat, sugarcane etc.) are preferred candidates in the plant kingdom for reuse. However, these by-products are littered in the open fields constituting environmental hazard by either burning them in open air or utilizing them for lesser applications such as; low-grade fuels, plant manure compost, stockbreeding floors, landfilling materials etc. Moreover, investigations (Mohanta et al., 2014; Nkayem et al., 2016) so far have revealed a high concentration of silicon dioxide (SiO_2) in monocot plant tissues which is a vital raw material in ceramic technology. Therefore, in order to comply with global best practices of managing waste materials, it is essential to resourcefully channel these agro-waste materials into the production of valuable ceramic components.

Considering the drawbacks experienced in other inorganic counterparts such as metals and polymers operating in aggressive chemical contacting applications, ceramics offer promising reliability in such medium owing to their high stability when subjected to chemical attacks. More so, with increasing sensitivity of researchers towards recent technological trends, porous ceramic materials have been extensively implemented as critical components in diverse hazardous industrial processes and pollution treatment technologies. In the meantime, studies (Striegler et al., 2018; Han et al., 2018) have shown both strong acidic and strong alkaline operating media as the major unfavorable service conditions encountered by the ceramic components.

Focusing on the wide-ranging separation technologies attainable, membrane filtration systems have emerged as one of the fastest growing alternatives to other conventional techniques owing to their cost effectiveness, ease of use and excellent separation efficiency. The ceramic membranes have been employed in acidic water treatment containing heteroatoms like Cl, S and P which are capable of oxidizing to form strong acids. Correspondingly, a recent study has showcased porous ceramics as having great potentials for soil salinity treatment in arid and semi-arid Mediterranean countries (Jalila et al., 2016). With the intent to seek advanced technologies for upgrading and optimizing existing industrial processes, ceramic heat exchangers and tubings are currently utilized in thermal storage facilities to avert hydrothermal corrosion hazards. In light of the aforementioned, the service environment pH for porous ceramic components should be of utmost concern to researchers.

So far, matrix grains incompatibility with processing additives and grain boundary defects have high disintegrating effect on ceramic components operating under corrosive media. Quite a number of studies (Curkovic et al., 2008; Muller et al., 2015) have highlighted impurities (silicon, magnesium etc.) originating from the starting materials as the primary cause for the dissolution of amorphous and impurity-rich grain boundaries in ceramic components. As a result, the morphology and mechanical properties of the structures are degraded. For now, corrosion studies for porous ceramic materials have been less comprehensive and the results documented thus far have shown a great level of inconsistency. Hence, considering the essentials of the current study, it is worthy of note to conduct an extensive corrosion study on porous ceramic materials operating under harsh service environment.

1.4 Aim and Objectives

This study aims at developing plain and Ni-reinforced porous alumina ceramics composites with agricultural wastes and nickel as the pore former and reinforcement respectively. In order to test the feasibility of the hypothesized porous ceramic systems, the following objectives are highlighted:

1) To analyze the mechanical and corrosion resistance properties of plain porous alumina ceramics as a function of porosity level and different agro-waste pore-forming agents.

2) To analyze the mechanical and corrosion resistance properties of Ni-reinforced porous alumina ceramics composites as a function of porosity level and different agro-waste pore-forming agents.

3) To develop Artificial Neural Network for predicting the mechanical and corrosion resistance properties of plain and Ni-reinforced porous alumina ceramics composites having formulations within the range of those employed in the experimental process through data training, validation and testing.

1.5 Scope of Study

In the current study, the mechanical properties evaluated for the plain and Ni-reinforced porous alumina ceramics composites include the hardness, tensile stress, tensile modulus and compressive stress. More so, the choices of pore former content (5, 10, 15, 20wt%) and nickel reinforcement content (2, 4, 6, 8wt%) utilized in developing the plain and composite samples respectively were made based on factors such as criterion sampling which serves as an ideal reference point from which the objectives of the study can be achieved. Furthermore, the discovery garnered during the trial fabrication process as well as the general decline observed in the densification, mechanical and corrosion resistance properties of the samples with increasing PFA or Ni reinforcement content support the choice of the sample formulations selected. More so, from the literature (Hammel et al., 2014; Mohanta et al., 2014; Nkayem et al., 2016), a similar trend made towards the choice of pore former content in the characterization of porous ceramics yielded information rich results which therefore justifies the interchange in sampling themes.

Meanwhile, the choice of the pore former content for developing the composites was determined having considered the experimental results obtained for the plain porous alumina ceramics. In particular, the mechanical and corrosion resistance properties of the plain porous alumina ceramics exhibited a sharp decline after exceeding the 10wt% PFA mark due to the intensified PFA agglomeration and the subsequent alumina grains dislocation prior the sintering process. Hence, the 10wt% PFA content was used to develop the Ni-reinforced porous alumina ceramics composites.

1.6 Thesis Outline

The first chapter in the thesis contains sub-sections that give a broad insight on the study as a whole. These include the background of research, the problem statement, the significance of study and the research objectives. The scope of study covers the mechanical properties investigated as well as the choices of pore former content and nickel reinforcement employed in developing the plain and Ni-reinforced porous alumina ceramics composites.

A critical review of relevant investigations on the various research themes in this study is presented in chapter two, including trends and overview of the important concepts in the present work. Overviews of theories, processing techniques and characterization methods are presented in order to aid the procedural steps embarked upon in this study towards meeting the standards set aside for the evaluation of the plain and Ni-reinforced porous alumina ceramics composites.

An experimental documentation of the steps and procedures employed in the data collection for the different aspects of this work are described in chapter three. Here, overview and discussions on the processing materials, sample processing techniques and microscopic/spectroscopic properties of the materials are presented. The various characterization theories and techniques including microstructural analysis, mechanical properties testing and corrosion resistance study for the evaluation of the developed plain and composite samples are discussed. More so, the methods employed for the development of artificial neural network models are highlighted in the chapter.

Chapter four contains the results obtained in the course of this study and the discussion of the data in relation with the research objectives and the existing investigations highlighted in Chapter two. The discussion for the results obtained for the Ni-reinforced porous alumina composites was made relative to the plain counterpart. More so, the predictive accuracy of the artificial neural network models (integrated by means of Graphical User Interface) which was trained with the results obtained for the plain and Ni-reinforced porous alumina ceramics composites is presented in the chapter.

In chapter five, the conclusion of the whole work and a summary of the results are presented, including major findings as well as the recommendation.

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