INVESTIGATION ON MECHANICAL PROPERTIES OF COCKLE SHELL COMPOSITE STRUCTURE UNDER TEMPERATURE AND AGING INFLUENCE

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ABSTRACT

Cockle shell is one of the marine organisms that can easily found in Malaysia. In this study, a ceramic material was fabricated from cockle shell and used sucrose as a binder. The cockle shell was crushed and milled into small size at around 160 µm -200µm. The cockle shell powder then was mixed and compacted with sucrose binder by ratio of 10, 20, and 30 wt%. The aim of this project is to find the relationship betweencockle shells powder with sucrose binder under influence of drying under oven, room temperature and aging process by months. To find the suitable temperature for the flexural test, compression tests have been done. The samples were placed in an oven at different temperatures of 50°C, 100°C, and 150°C to test the compressive strength.The compression test result shows that the samples with 78wt% of cockle shell at 150°C has the highest strength compare to 68wt%, 88wt% of cockle shells at different temperatures 50°C and 100°C. The drying process was implemented in two methods which heated the samples for an hour at 150°C and another method was the sample placed for drying under room temperature for 24 hours before the flexural test. The effect of binder percentage, drying method and aging process on density, porosity, flexural test, microhardness test, and morphology were investigated. From based on compression test, another set of cockle shell with sucrose binder by same ratio were prepared for flexural test. Density, porosity and flexural test result shows that the samples with oven drying produced better strength compared with samples dried at room temperature for 1 day, 1 month, 3 months and 9 months and the highest strengthwas obtained by sample with 78wt% cockle shell dried in oven. The finding that integrated, samples with 78wt% of cockle shell at 150°C has the best characteristics under physical and mechanical properties.

ABSTRAK

Kulit kerang adalah salah satu organisma marin yang mudah didapati di Malaysia. Dalam kajian ini, bahan seramik dibuat dari kulitkerang dan digunakan sukrosa sebagai pengikat. Kulit kerang telah dihancurkan dan dijadikan dalam saiz kecil 160µm-200µm. Seterusnya, serbuk kulit kerang di campur dan dipadatkan dengan pengikat sukrosa dengan nisbah 10,20, dan 30%. Tujuan projek ini adalah untuk mencari hubungan antara serbuk kulit kerang dengan pengikat sukrosa di bawah pengaruh pengeringan dan proses penuaan. Untuk mencarisuhu yang sesuai untuk ujian lenturan, ujian pemampatan telah dilakukan. Sampel diletakkan di dalam oven pada suhu yang berbeza 50°C, 100°C, dan 150°C untuk menguji kekuatan mampatan. Hasil ujian mampatan menunjukkan bahawa sampel dengan 78wt% spesimen pada 150°C mempunyai kekuatan tertinggi dibandingkan dengan 68 wt%, 88wt% spesimen pada suhu yang berbeza 50°C dan 100°C. Proses pengeringan telah dilaksanakan dalam dua kaedah iaitu memanaskan sampel dalam ketuhar selama satu jam pada 150°C dan satu lagi kaedah ialah sampel yang diletakkan dalam suhu bilik selama 24 jam sebelum ujian lentur danmampatan. Kedua, dalam proses penuaan, sampel diletakkan di bawah suhu bilik selama 1 bulan, 3 bulan dan 9 bulan. Pengaruh peratusan pengikat, kaedah pengeringandan proses penuaan terhadap ketumpatan, keliangan, ujian lenturan, ujian mikrohardness, dan morfologi diselidiki. Dari berdasarkan ujian mampatan, satu lagi set specimen dengan pengikat sukrosa dengan nisbah yang sama disiapkan untuk ujian lenturan. Hasil ujian ketumpatan, keliangan dan lenturan menunjukkan bahawa sampel dengan pengeringan ketuhar menghasilkan kekuatan yang lebih baik berbanding dengan sampel yang dikeringkan pada suhu bilik selama 1 hari, 1 bulan, 3bulan dan 9 bulan dan kekuatan tertinggi diperoleh dengan sampel dengan kulit kerang 78% berat dikeringkan di dalam ketuhar. Penemuan disatukan sampel dengan 78wt% yang kerang pada suhu 150°C mempunyai ciri-ciri terbaik dalam sifat fizikal dan mekanikal.

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS AND ABBREVIATIONS

$FESEM$	-	Field-Emission Scanning Electron Microscope
η	-	Thermal efficiency
DCAM	-	Department of Civil Aviation of Malaysia
ASTM	-	American Society for Testing and Materials
WT%	-	Weight Percentage
σ	-	Flexural Strength
SEM	-	Scanning Electron Microscope
L	-	Length
W	-	Width
T	-	Thickness
MPa	-	Megapascal
µ	-	Micron
g	-	Gram

CHAPTER 1

INTRODUCTION

1.1 Background of Study

One of the most basic issues in a defensive system is protective material. Protection induces a move in soldier's internal paradigm and contributes to multiple psychological endurance to move strongly in the battlefield, creating a significant force-multiplier effect (Yadav, Naebe, Wang, & Kandasubramanian, 2016). Armor plays a major role in protecting warriors. The introduction of new materials and improvement in the materials that are already used to construct armour have led to better protection and reduction in the weight of the armour. During World War I (1914-1918), the army involved in the war used soft body amour which was manufactured from silk (Bellis, 2020). In World War II (1939-1945), the material of the protective covering has been developed, and they tend to use 'flak jacket' made from ballistic nylon was used in order to protect their body or vehicle from bullet during battle (Chen & Zhou, 2016).

The history of enhancing protection while decreasing armour weight has been a remarkable success story. New material decisions such as ceramic, polymer, polymer fibre and lower density metal have considerably reduced the weight of the armour required to protect people and vehicles over the past half-century (Bhatnagar & Asija, 2016). Protection is effectively known to allude to a defensive coverage that can protect the body, build or cars from harm or assault. Instead of commonly used in the war zone, the protective material connected to our daily lives, for instance, hockey head protector and bike cap, which could potentially avoid head damage.

The evolution of the material as part of plate armour continues to change from Kevlar steel, ceramics and materials that can make a better impact and benefit users

(Crouch, 2019). In order to get suitable and easier found and fabricated products to produce protective material, a study has been done to distinguish if cockle shells can be one of the fundamental sources to produce protective materials due to the properties of cockle shells that have unique mechanical properties (Bharatham et al., 2014). The cockle shell is composed of approximately 96% CaCO3, with the other components include of organic substances and other oxides such as SiO2, MgO, and SO3. Calcite, aragonite, and vaterite are the three polymorphs of CaCO3 Nonetheless, calcium carbonate aragonite polymorph has recently emerged as one of the most popular targets for exploration among several scientists, particularly in the biomedical and pharmaceutical fields. Further to that, due to its unique properties for a variety of medical uses, aragonite polymorph of calcium carbonate is shown to have high potential as just a better biomedical substance to be incorporated, settled, and supplanted by bones. Aragonite is less thermodynamically stable than calcite at room temperature and pressure, it is well known to be denser than calcite, sensitive to variations in temperature, comprising some morphological varieties, having high mechanical strength, and biocompatible and biodegradable.(Liyana, Abd, Hussein, Abu, & Zakaria, 2017)

One of the most important and common species of mollusc in the aquaculture industry in our country is cockle or its scientific name, *anadara granosa.* Locally, it is known as ‘kerang’, and it is a famous local delicacy. Cockle is one of the foremost abundant maritime potentials within the province since it can be effectively found in nearly all coastal zones. Along the west coast of Peninsular Malaysia including Johor and Pahang, 57,544.40 tons of cockles were harvested according to the statistics by the Department of Fisheries in the year 2011. From this massive value, it is clear to us on how great the cockle industry is, which also means that more shell wastes are generated (Olivia et al., 2017).

Most of the time, cockle shells are thrown away and considered as wastes once eaten. From this massive cockle industry, it only means that more cockle shells will be thrown to the environment. This irresponsible action causes environmental degradation and pollution because the dumped cockle shells will take a long period of time to completely decay. (Othman et al., 2013). This is because, calcination is an endothermic reaction which requires high temperature and low carbon dioxide partial pressure to proceed (Mohamed, Yusup, Quitain, & Kida, 2018). It also indicates that the active and lucrative industry has resulted in a significant amount of waste shells (Boey et al., 2011). So, as a responsible citizen of Mother Earth, people should take the initiative to support eco-friendliness. This study aims to implement the concept of green technology in engineering by using cockle shells as alternative material.

Mechanical and thermal properties attributed to crystalline and elemental structures are what cockle shells are notably known for. Generally, shells are composed of calcium carbonate which results in high strength, low mass and low coefficient of heat conductivity (Kuo et al., 2013). Throughout the years, various researchers from all around the world have conducted studies to put cockle shell wastes to good use. An example of study done by Hoque et al. (2013) has investigated the use of cockle shell wastes as bio-ceramics in bone application. The CaCO3 bioceramic powder was successfully synthesized from cockle shells and characterized by SEM, EDX, XRD and FT-IR. Calcium carbonate (CaCO3) bioceramics derived from Malaysian cockleshells have high potential in bone tissue engineering applications (Hoque, 2013).

Crushed cockle shell powder is utilised as a source of calcium oxide in catalysing a trans-esterification reaction to produce biodiesel (methyl esters) (Boey et al., 2011). Finally, a study is done on the use of cockle shell wastes as substitutes for aggregates in construction materials (Muthusamy et al., 2012). By observing all these findings, it suggests that cockle shells have the potential for other uses as well.

1.2 Problem Statement

After some explorations have been made to discover new wellspring of material in ceramic coating for car, cockle shells are picked as one of the trial materials because of its mechanical properties that can possibly be discovered. A number of researchers have developed cockle shell powder in many productions such as concrete, bone repairing and as a catalyst in biodiesel production (Olivia et al., 2015; Awang-Hazmi et al., 2007; Boey et al., 2011). Considering these approaches, a study is conducted to investigate the mechanical and physical properties of cockle shell.

The quantity of cockle shell wastes has risen significantly in recent years, and if this trend continues, it will result in serious environmental issues. Consequently, these are left untreated and dumped irresponsibly, cockle shells may also produce unpleasant odour (Mustakimah Mohamed, Suzana Yusup, 2012). Therefore, this research is carried out to allow us to maximize the use of cockle shells as an environmentally friendly alternative material under focus on sustainable development goal 12; Responsible consumption and production. Achieving economic growth and sustainable development allows us to reduce our ecological footprint as a matter of urgency by improving the way people produce and consume goods and services. Reusing this waste material is equally important, as is supporting developing countries to move towards more sustainable patterns of consumption by 2030 (Sustainable Development Goals, 2020). In addition, research shows that shells can improve the strength of the other ingredients when used as an aggregate. (Paper et al., 2012). Moreover, the shell structure is a composite substance which is naturally formed. (Olivia et al., 2017).

Therefore, this project presents study focuses on the mechanical properties of cockle shell structure which experimental results on the flexural behaviour of cockle shells with binder. There are 3 important parameters used, namely sucrose binder percentage, type of drying process and aging process. Then, the cockle shell ceramic specimens will be tested according to the ASTM standard. Finally, the flexural behaviours are discussed in terms of these 3 parameters with the assistance of density, porosity, hardness and Scanning Electron Microscopy (SEM).

1.3 Aim

To evaluate the relationship between cockle shell powder with sucrose binder, temperature influence and aging process towards strength.

1.4 Objectives

The objectives of the study are to:

i) Determine the suitable oven curing temperature for cockle shell specimen under compression test with different percentage of sucrose binder.
ii) Examine the physical and mechanical properties of cockle shell with different percentage of sucrose binder under drying and aging process.
iii) Evaluate the influence of temperature and aging process under different percentage of cockle shells and sucrose binder.

1.5 Scope of Study

The scopes of the review undertaken include:

i) The main materials used for this project are cockle shell powder ranged in between 160µm - 200µm and sucrose.

ii) The chosen parameters are:

   a) Sucrose as binder mixed with cockle shell powder is tested at different percentage such as 10wt%, 20wt% and 30wt%.

   b) The different percentage of cockle shell specimen are placed in oven at 50°C, 100°C and 150°C for one hour and dried in room temperature for one day for compression test.

   c) Two types of drying methods used which are drying samples in oven curing at 150°C for 1 hour continuous with one day drying under room temperature and another set was dried under room temperature for one day for density test, porosity test, flexural test, hardness test and SEM.

   d) Aging process where samples are placed under room temperature for 1 month, 3 months and 9 months for density test, porosity test, flexural test, micro hardness test and SEM.

iii) Perform physical properties test:

   a) Density analysis by using Mettler Toledo Density Kit

   b) Porosity analysis by using Mettler Toledo Density Kit

iv) Perform mechanical properties test:

   a) Compression test by using Universal Test Machine in accordance with ASTM C773-888.

   b) Flexural test by using Universal Test Machine in accordance with ASTM C1161-13.

   c) Vickers micro hardness test by using Shimadzu HMV series Micro Vickers Hardness Tester in accordance with ASTM E384.

v) Perform morphology test:

   a) Determine the fracture surface of samples by using Field Emission Scanning Electron Microscope (FESEM).

1.6 Significance of study

The main objective of this study is to determine the strength of natural wastage of cockle shells using specific tests such as flexural strength test based on the test standard ASTM C1161-13. The specimens are compacted with cockle shell powder with different percentage of sucrose binder (i.e. 10wt%, 20wt% and 30wt %), then dried in 3 different methods. Flexural strength test is intended to identify the maximum strength that can be accepted by the resulting sample-based shells. Thus, this research represents an important initial step to introduce cockle shells as the new alternative material, where initially it goes down as just a waste, and due to this study, it will never be a waste anymore.

1.7 Summary of Research

This research about to study the sourcing alternative sustainable materials for protective layer in order to minimise over-reliance on natural resources. The use of solid waste, such as cockle shells, makes the process more environmentally friendly, green, and cost-effective. A promising solution to the challenge of cockle shell waste management involves utilising cockle shell as protective layer. In this research, the first present the compression test conducted before the other test to find the suitable temperature for oven curing. The samples were used in compression test only after go through oven at 50°C, 100°C and 150°C for one hour and dried in room temperature. This is followed by, analysis of the physical and mechanical properties of cockle shell at different percentage of sucrose binder and drying process with different time duration. Based on the experiment, specimen with 78% of cockle shell with 150°C oven curing for 1 hour gives the best result in terms of flexural test, microhardness test, compression test, density test and porosity test. This sample is summarised as best and optimum range sample for further studies.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter contains a brief explanation about project based in literature review. It contains earlier research on the involvement of shells in some similar aspects. Detailed introduction about the basic fundamentals are involved in this study, such as cockle shells, binders, the three-point flexural test, hardness test, compression test, density, porosity and Field Emission Scanning Electron Microscope (FESEM).

2.2 Armour Protection

Armour protection is one of the most significant elements of survival. Protection creates a change in a soldier's inner paradigm and leads to multiple psychological stamina to move fearless in the battle field, which generates a massive force-multiplier effect. Throughout the history of warfare, material technologies have a significant impact on the capacities of land-combat forces. Armour materials have progressed by improved metal structures, advanced development and lightweight composite materials (low areal density). Advances in ceramic systems have further improved quality (Vemuri & Balakrishna, 2011).

Similarly, the development in the growth of explosive reactive armour has produced an effective armour system against all modern high explosive anti-tank ammunition and missile threats to armoured vehicles. However, to obtain armour efficiency that exceeds the current light combat vehicles and main battle tanks for new vehicle systems, weighing considerably less than the current combat vehicles, developments in new armour components, systems and survivability technologies are expected (Vemuri & Balakrishna, 2011).

A survey conducted by Vemuri and Balakrishna (2011) has shown that advanced armour material is developed from steel armour, titanium armour and combination of ceramic and polymer armour (Vemuri & Balakrishna, 2011). The evolution of armour materials is summarised in Table 2-1.

Table 2-1: The evaluation of armour materials (Vemuri & Balakrishna, 2011)

Materials	Advantages	Disadvantages
Steel amour	This can be accomplished by developing new alloys and adjusting appropriate heat treatments. On the other side, metal gives better strength and toughness combinations with lesser concentration of hardness.	Heavy weight
Titanium armour	Titanium has high strength to weight ratio, better corrosion resistance, and great ballistic performance compared to aluminium alloys and metal.	Titanium is formed by adiabatic shear band, which can cause spalling.
Ceramics and Polymer armour	Ceramics have the appealing characteristics of greater hardness, reduced density, greater modulus with some flexural strength and toughness of fracture.	The mechanical properties of ceramics cause the penetrator to fracture, and the ability of the rear layer to catch the projectile debris and the damaged ceramic material.

As the evolution of material used as part of the plate armour happens, from Kevlar steel, ceramics and materials, which provide consumers better impacts and benefits. Today, no single material is capable of defeating a wide range of threats effectively, and a wide variety of armors must therefore be created. In order to get friendlier and easier products to produce protective material, a study is conducted to distinguish either cockle shells can be one of the fundamental sources to produce protective material due to the properties of cockle shells that have unique mechanical properties.

2.3 Natural Materials

Natural materials may demonstrate great combinations of rigidity, low weight, strength and toughness, also unmatched by human made materials (Barthelat et al., 2009). A huge effort was therefore made in the material science community over the last two centuries to explicate the microstructure and mechanisms behind these mechanical performances in order to reproduce them in artificial materials (Barthelat, 2007). This design method, called biomimetic, has now begun to produce materials with notable characteristics. The first step in this biomimetic approach is to recognize the performance of materials in natural materials, along with fundamental knowledge of the processes behind all these outcomes. (which have been greatly accelerated by recent techniques such as scanning probe microscopy) (Barthelat & Espinosa 2007).

The mechanical performance of natural materials is demonstrated in Figure 2-1, a material properties map for a selection of natural ceramics, biopolymer and their composites (Wegst & Ashby, 2004). The top left corner of the map demonstrates soft and tough materials such as skin, with a mechanical behaviour similar to natural elastomers. The lower right corner of the map demonstrates stiff but brittle minerals such as hydroxyapatite or calcite. Most hard biological materials incorporate minerals into soft matrices, mostly to achieve the stiffness required for structural support or armoured protection (Currey, 1999).

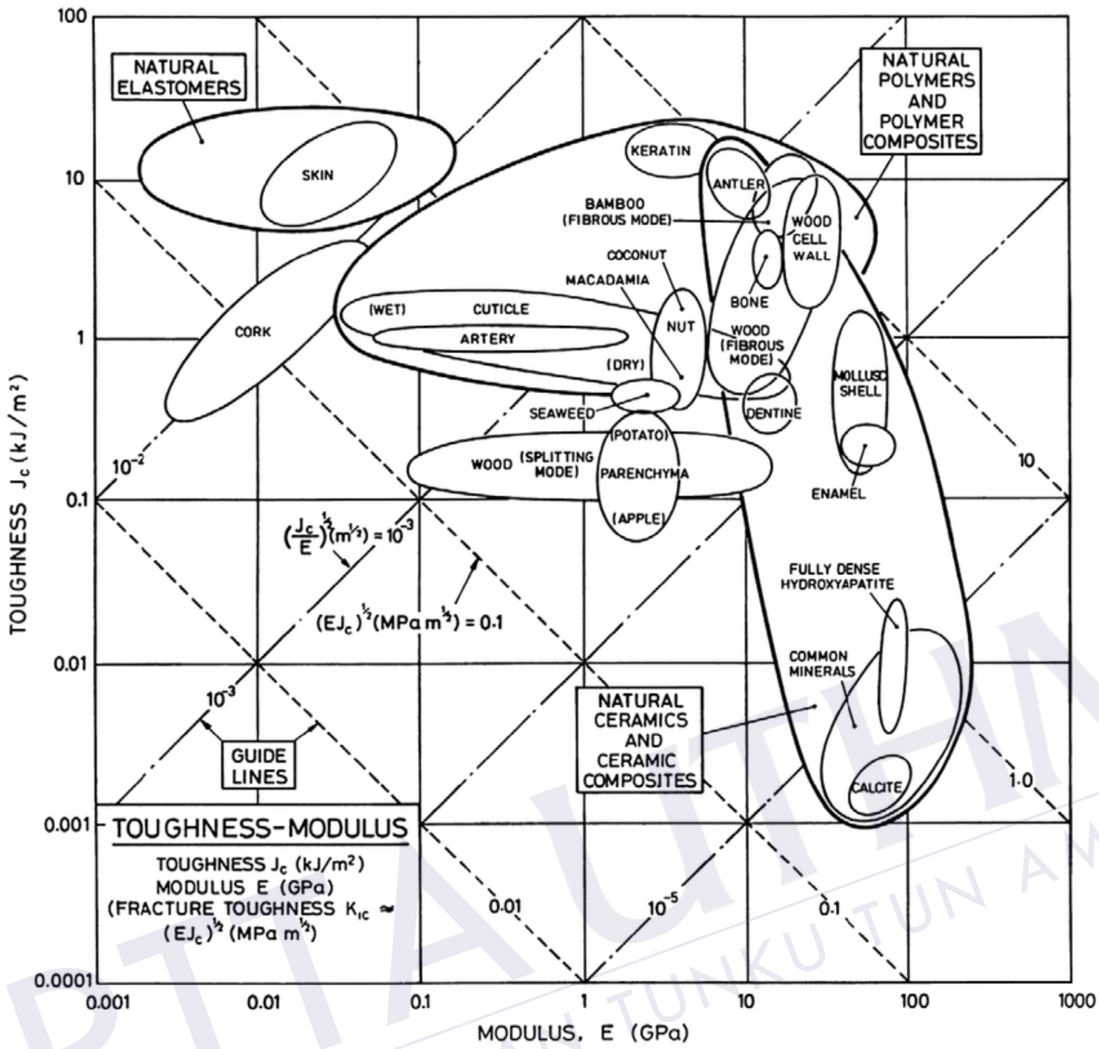

Figure 2-1: Material properties map for a variety of ceramics, soft natural tissues and their composites.

Wegst and Ashby (2004).

The effect of combining soft proteins with rigid minerals can be seen in Figure 2-1, a chart of a material properties natural ceramics, biopolymers and their composites. (Wegst & Ashby, 2004). The modulus is a stiffness measure represented on the horizontal axis, while the toughness is represented on vertical axis.

The vertical axis (toughness) is a measure of the ability of the material to resist cracking, while the horizontal axis (modulus) is a measure of the stiffness of the material. The upper left of the diagram is the domain of the ‘soft’, natural elastomers such as skin; they are compliant and hard materials that tear rather than crack.

The bottom right is the hard domain, with minerals not only stiffer than elastomers, but also much more fragile (low toughness). The upper right of the diagram in Figure 2-1 is

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2. **R. Anpalagan**, K. Kamarudin, Muhammad Iqbal Haikal Roslan, M. N. M. Hatta (2019) .Mechanical Properties of Seashell Structure under Different Drying Temperature.
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