SYNTHESIS OF ALUMINOSILICATE FROM IMPREGNATION OF RICE HUSK

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ABSTRACT

This study was carried out to produce aluminosilicate from impregnation method. In this study rice husk was impregnated into with aluminum nitrate solution. Rice husk act as silica source, while aluminum nitrate contribute to alumina source in synthesizing aluminosilicate. The rice husk was impregnated with different concentration of aluminum nitrate. From the result obtained at temperature 200°C till 600°C in 2 mol concentration of aluminum nitrate, there were no peaks formed. This is because crystallization of mineral does not occur at low temperature. So, at temperature of 800°C there have several peaks occur at 2θ anlge of 35.00° to 40.00°, 45.00° to 50.00 to 65.00° to 70.00°. that can be observe but the peak intensity was too low for XRD to analyzed it. Andalusite do not formed at this temperature and concentration. For AS2 sample, at 200°C till 600°C there have only one observable peak occur at 2θ angle around 45.00° ,the mineral present cannot be identified. At this temperature and concentration andalusite still does not formed. Andalusite and alumina was formed at AS2 800℃ sample. Its occur at 2θ angle of 19.280°, 23.172° and 25.165°. For XRD pattern of alumina it's occur at 2θ angle of 16.441°, 20.058 and 21.705°.

ALUMINASILIKA SINTESIS MENGGUNAKAN IMPREGNETASI OLEH SEKAM PADI

ABSTRAK

Kajian ini telah dijalankan untuk menghasilkan aluminasilika daripada sekam padi melalui proses impregnetasi menggunakan aluminum nitrik yang berbeza kepekatan. Sekam padi bertindak sebagai punca silica manakala aluminum nitric bertindak sebagai punca aluminum oksida. Daripada keputusan yang diperolehi, pada suhu 200°C hingga 600°C dalam kepekatan 2 molar tiada puncak yang dapat diperhatikan. Ini kerana kristalisasi hanya berlaku pada suhu yang tinggi iaitu lebih dari 1000°C. Bagi suhu 800°C terdapat beberapa puncak yand dapat dilihat berlaku di 2θ anlge berdekatan 35.00° hingga 40.00°, 45.00° hingga 50.00°, 65.00° hingga 70.00° yang boleh diperhatikan tetapi intensiti puncak tersebut terlalu lemah untuk dianalisa. Andalusita tidak terhasil pada suhu dan kepekatan ini. Untuk sampel bagi AS2 di suhu 200°C hingga 600°C hanya terdapat satu puncak yang dapat dilihat terjadi di 2θ angle berdekatan 45.00°. Mineral yang wujud pada puncak itu tidak dapat dihasilkan. Pada suhu dan kepekatan ini andalusita tidak dapat dihasilkan. Andalusita dan aluminum oksida terhasil pada sampel AS2 pada suhu 800°C. Ia terhasil pada 2θ sudut 19.280°, 23.172° dan 25.165°. Untuk XRD analisa untuk aluminum oksida ianya berlaku pada 2θ sudut 16.441°, 20.058 dan 21.705°.

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LIST OF SYMBOLS, UNITS AND ABBREVIATION

FTIR	Fourier Transform Infrared Spectroscopy
XRD	X-ray Diffraction
℃	Degree Celsius
%	Percentage
&	And
ml	Millililter
g	Gram
IR	Infrared
g/mol	Gram per mol
cm^{-1}	Per centimeter
kg	Kilogram
M	Molar
RH	Rice Husk
LH	Leached Husk
$C_5H_8O_{10}$	Hemicellulose
$C_7H_{10}O_3$	Lignin
NaOH	Sodium Hydroxide
-COOH	Carboxylic acid
SiO_2	Silica oxide
OH	Hydroxyl group
HF	Hydrofluoric Acid
$Al(NO_3)_3$	Aluminium Niitrate
C=C	Alkene

CHAPTER 1

INTRODUCTION

1.1 Research Background

Recently, there has been a lot of research about the uses of rice husk that give a lot of advantage in ceramic industry. Rice husk is an abundantly waste material in rice producing industry. The usage of rice husk in raw form or in ash form, both has many advantages. Rice husks are the natural sheaths that are formed on the rice grains during growth and are removed during the refining of rice; thus, it does not have commercial interest (Real *et al.,* 1996). The main components of rice husk are cellulose ($C_5H_{10}O_5$), lignin ($C_7H_{10}O_3$), hemicelluloses ($C_5H_8O_{10}$) and primary ash (SiO_2) but it differ with climate and geography (Feng *et al.,* 2004). The structures of rice husk are composed of dentate rectangular element which they are composed mostly silica coated with thick cuticle and surface hairs. The mid region and inner epidermis contain silica (Bronzeok, 2003). Silica contain in rice husk is very high up to 75% - 95%. Amorphous silica is concentrated at the surface of the rice husk and not within the husk itself (Jauberthie, *et al.,* 2002).

Most of the husk from the milling is either burnt or dumped as waste in an open fields and a small amount is used as fuel for a boiler, electricity generator, bulking agent for compositing of animal manure (Bronzeok, 2003). Pure silica can be obtain by acid leaching process of rice husk (Chang *et al.,* 1991). After burnt, silica is then converted to the crystalline form of silica (Chandrasekhar *et al.,* 2003). Research by Real *et al.,*(1996)

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It was then washed thoroughly with distilled water followed by treatment with dilute ammonia solution to remove the traces of acid (Chang *et al.*, 2001). To obtain high purity of silica is important because, silica is one of the raw materials for traditional ceramic.Ceramic come from the Greek words mean 'burnt stuff'. Ceramic always composed of more than one element. The desirable properties of ceramic usually achieved through a high temperature heat treatment process or known as firing (Rack, 2002). Ceramic has different properties depends on the type of ceramic produced. Chemical properties of ceramic include the stability, instability, and the reactivity of material under the influence of variable such as temperature and or severe atmosphere.

The physical properties of ceramic referred to their thermal, electrical, magnetic and optical properties of the ceramic (Wiley, 2002). There are three basic categories of ceramic, which are, traditional ceramic, new ceramic and glass ceramic. Aluminosilicate is the naturally occurring mineral and also can be synthesized chemically. The basic compound of the aluminosilicate consists of aluminum, silicon and oxygen, an additional compound likes cation that present in the aluminosilicate compound will formed minerals such as garnets, topaz, kaolinite, feldspar and many others (Pinkas, 2005). Aluminosilicate may have different physical properties due to the different arrangement of atom or molecules in the compound (Petersen *et al.*, 2013).

1.2 Objective

The objectives of this study are:

i. To synthesize aluminosilicates compound by impregnating the rice husk with aluminum nitrate.

ii. To characterize the aluminosilicates compound formed using x-ray diffraction (XRD) and fourier transform infrared spectroscopy (FTIR).

1.3 Scope of study

This study focused on the synthesis of aluminosilicates compound using waste mineral which is rice husk as the silica source. The silica is prepared from the acid leaching of rice husk. The leached rice husks are impregnated with aluminum nitrate solution at different temperature from 200°C to 800°C with the different concentration 2M, and 4M of aluminum nitrate solution. The variable in this study are the temperature and the concentration of the aluminum nitrate solution. The aluminosilicates compound produce are then characterized using X-ray diffraction (XRD) and fourier transform infrared spectroscopy (FTIR).

CHAPTER 2

LITERATURE REVIEW

2.1 Ceramic

The first use of functional pottery vessels is thought to be in 9,000 BC. These vessels were most likely used to hold and store grain and other foods. Ceramic is defined as the hellenic word *keramos* meaning 'fired soil' in Greek, on the other hand called ceramics. It is defined as the art and science of making material and product of non-metallic inorganic substance (Geiger, 2001). Ceramic is prepared by the action of heat and subsequent cooling (Gritlahare, 2009).

Theoretically, the strength of ceramics should be higher than metal because of the covalent and the ionic bonding types are stronger than metallic bonding. However, metallic bonding allow for slip, the basic mechanism by which metal deforms plastically when subjected to high stresses. Bonding in ceramic is more rigid and does not permit slip under stresses. The inability to slip makes it much more difficult for ceramics to absorb stresses. Physical properties of ceramics are density, generally, ceramic are lighter than metals and heavier than polymer. The melting temperature of ceramics is higher than for most metal and some ceramic are decomposed rather than melt (Wiley, 1997).

Research by Peter (2009) said, the properties of ceramic materials are depends on the types of atoms present, the types of bonding between the atoms and the way the atoms are packed together. The type of bonding and structure helps to determine what type of properties a certain material have. Ceramics usually have a combination of stronger bonds called ionic usually occurs between metal and non-metal compound that involves the attraction of opposite charges ion, when electrons are transferred from the metal to non-metal compound and covalent bond occurs between two non-metals compound and involves sharing of atoms. The strength of an ionic bond depends on the size of the charge on each ion and on the radius of each ion.

The greater the number of electrons being shared, is the greater the force of attraction, or the stronger the covalent bond. These types of bonds give high elastic modulus and hardness, high melting points, low thermal expansion, and good chemical resistance. On the other hand, ceramics are also hard and often brittle unless the material is toughened by reinforcements or other means, which leads to fracture.

According to Peter, (2009) in his research, also state that the metals have weaker bonds than ceramics, which allows the electrons to move freely between atoms. This type of bond results in the properties called ductility, where the metal can be easily bent without breaking, allowing it to be drawn into wire. The free movement of electrons also explains why metals tend to be conductors of electricity and heat.

Plastics or polymers of the organic type consist of long chains molecules which are either tangled or in ordered at room temperature. Because of Van Der Waals force between the molecules are very weak, polymers are very elastic like a rubber band, can be easily melted, and have low strength. Like ceramics, polymers have good chemical resistance, electrical and thermal insulation properties. They are also brittle at low temperatures.

Research by Singh (2009), state that ceramics possess chemical, mechanical, physical, thermal, electrical, and magnetic properties that distinguish them from other materials likes metal and plastics. Manufacturers customize the properties of ceramics by controlling the type and amount of the materials used to make them. Chemical properties of the industrial ceramic are primarily oxides compound of oxygen, but some are carbide compounds of carbon and heavy metal, nitrides compound, borides and silicides compound. For example, aluminum oxide can be main ingredient of ceramic such that the important alumina ceramics contains 85% to 99% aluminum oxide.

Primary components such as the oxides can also be chemically combined to form complex compounds that are the main ingredient of the ceramic. Ceramic are most resistant to corrosion than plastic and metal. Ceramic generally do not react with most liquid, gases, alkaline and acids. Most ceramic have very high melting point.

In ceramic industry, the manufacturing of ceramic is produce by several steps which is, material preparation, die pressing, isostatic pressing, extrusion, injection moulding, sintering, tape casting, polarizing, alloy forming, melt-spun forming, roll forming, chemical vapour deposition (CVD), ion beam and RF plasma deposition (Morgan, 2009). Ceramic materials are utilized for many difference applications in a variety of field. There are many types of ceramic, each with unique individual properties. For example, glass ceramic, porous ceramic and many other types of ceramic.

Applications of ceramic are earthenware, which is often made from clay, quartz and feldspar. Other applications of ceramic are knife blade. The blade of ceramic knife will stay sharp for much longer than the steel knife, although it is more brittle. Second application is ballistic armored vest to repel large-caliber rifle fire. These vests are made up of alumina and boron carbide.

2.2 Aluminosilicate

Silicon and aluminum are the second and third most abundant elements in the Earth crust but they are never encountered in the nature in their elemental form. Instead they combine with oxygen and form a multitude of aluminosilicate mineral such as garnets, topaz, beryl, pyroxenes, amphiboles, kaolinite and many other (Pinkas, 2006).

Aluminosilicate minerals are minerals that composed of aluminum, silicon and oxygen. The most basic form of aluminosilicates mineral has the basic molecular formula of Al_2SiO_5. Rock minerals that have the same molecular formula as aluminosilicates are known as siliminate, andalusite and kyanite (Whitney, 2002).

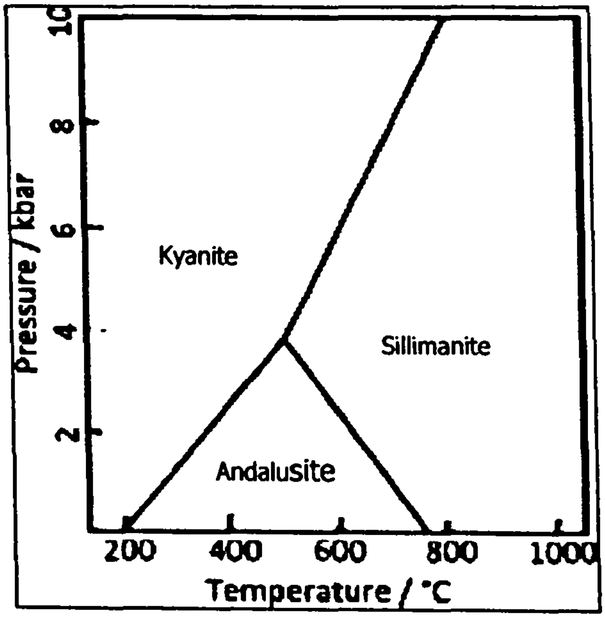

Figure 2.1 Phase diagram of Al_2SiO_5 by Whitney, (2002).

Kyanite mineral is a polymorph mineral that shares the same chemistry but a different crystal structure with other mineral likes andalusite and sillimanite. Kyanite is an attractive mineral that has a near sapphire-like blue color. It has nearly unique characteristic. It has wide variation in hardness in the same crystal face. Other minerals usually have variable hardness on different crystal face due to a different concentration and orientation of the atoms in the structure. Kyanite is a refractory material, it has high melting point, low thermal conductance and maintain its strength up to about 1100° C (Hobart, 2013).

Andalusite mineral are important raw materials for the production of superior grade high-alumina refractories for both the ferrous and the non-ferrous industries. The relative proportions in which these sister minerals are used world-wide depends to a large degree on their availability. However, anadalusite has some important advantages that are associated with its low relative density, its related lower volume expansion on being heated, and the fact that it can be produced by a relatively simple mining and concentration procedure at its natural grain size.

The Increasingly severe service to which refractories are now subjected in iron and steel plants has given rise to a change from fireclay materials to materials of higher alumina content. Low costs, energy-saving requirements, firebrick production techniques and the ultimate chemical, physical and dimensional characteristics of the brick combine to make andalusite the logical choice as a raw material to provide the additional alumina (Overbeek, 1989).

For sillmanite mineral, it is naturally occurring anhydrous aluminium-silicate polymorphs with the chemical formula Al_2SiO_5. The industrial use of sillimanite minerals is especially related to their unique chemical composition, stability at high temperatures and transformation to mullite rich aggregates utilized as refractory material (Ihlen, 2000). Alumino-silicate polymorphs with a stoichiometric composition of 62.93% Al_2O_3 and 37.07% SiO_2. The crsytal lattices give room for only limited substi tution by Fe, Mn, Ti and Cr; mainly in andalusite and kyanite. The bladed, needle-shaped and long prismatic crystals show variable colours in mainly shades of grey, pink, yellow and blue (Deer *et al*,. 1966).

Some of these minerals form the basis of many types of clay, particularly the fine white clay mineral known as kaolin and are used in ceramic manufacturing. There are other types of more complex aluminosilicates minerals that are found in nature have different chemical formulas and other element as part of their structure. For example, sodium aluminosilicates minerals are aluminosilicates that contain sodium atom. Others may contain potassium, calcium, sulfur, lithium or even combination of two or more element (Peterson *et al.* 2013).

Aluminosilicates mineral has different properties depends on the element exist in the aluminosilicates compound. According to Chavis (2013), hydrated aluminosilicates are known as zeolite. Pores in zeolite minerals are microscopic making them become main compound for absorbing small molecules of substance or liquid. In nature, the zeolite mineral are are created when volcanic ash and rock interact with base such as alkaline liquids. Natural zeolites usually are not very pure due to the contamination with other elements such as metal and crystal.

Other examples of aluminosilicate combined with other element which give big advantage to the ceramic industry are lithium aluminosilicates. This type of aluminosilicate produced glass ceramic that have high resistance to heat. The most important system is the Li_2O x Al_2O_3 x $nSiO_2$-System (LAS-System). The LAS-system mainly refers to a combination of lithium-, silicon-, and aluminum-oxides with additional components such as glass-phase forming agents such as Na_2O, K_2O and CaO and refining agents. As nucleation agents most commonly zirconium (IV)-oxide in combination with titanium(IV)-oxide is used. The most interesting properties of these glass-ceramics are their thermomechanical properties. Glass-ceramic from the LAS-System is a mechanically strong material also can sustain repeated and quick temperature changes up to 800–1000 °C (Hummel, 1951).

Aluminosilicate minerals are widely used as a raw material that has found a wide application in industry and material science. Among others, clay minerals are used as a suitable source for the preparation of aluminosilicate inorganic polymers (AIP) that exhibit increasing industrial interest, due to the low energy requirements of their manufacture and their promising mechanical properties such as compression strength, heat and chemical resistance. Other application of aluminosilicates is providing an energy-saving and environmentally friendly approach to synthesize zeolite Y from natural aluminosilicate minerals without the involvement of aluminum and silicon containing inorganic chemicals was developed. When used as a fluid catalytic cracking catalyst, the resulting zeolite Y exhibited an outstanding catalytic cracking performance. One of the useful product produce from aluminosilicate is aluminosilicate wool (ASW). It is also known as refractory ceramic fibre, are armophous fibre produced by melting a combination of Al_2O_3 and SiO_2.

2.3 Rice husk

Rice husk is one of the most widely abundant agricultural wastes in many rice producing country around the world. Globally, approximately 600 million tons of rice paddies are produced each year. On average 20% of the rice paddy is husk, giving an annual total production of 120 million tons (Giddel *et al.*, 2007). Rice husk contains 75% - 90% organic matter such as cellulose, lignin and rest mineral component such as silica, alkali and trace element (Madhumita *et al.,* 2009).

Rice plant is one of the plants that absorbs silica from the soil and assimilates it into its structure during the growth. Rice husk is the outer covering of the grain of rice plant. Rice husk (RH) is the waste material obtained from agricultural by-product. It constitutes about 20% of the weight of rice. The main constituents of rice husk are 50% cellulose ($C_5H_{10}O_5$), 25-30% lignin ($C_7H_{10}O_3$), 15-20% hemicelluloses ($C_5H_8O_{10}$) and primary ash (SiO_2) but it differ with climate and geography (Feng *et al.*, 2004).

Rice husk ash (RHA) is obtained by burning of rice husk. Upon burning, cellulose and lignin are removed, leaving behind silica ash. High reactive RHA is obtained when RHA is burnt under controlled conditions. This RHA contains a high content of silica in the form of non-crystalline or amorphous silica up to 95% (Della *et al.*, 2002).

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