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Oxidation of Phenol using Silica Nanoparticles from Rice Husk Ash: Effect of Solvent

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DECLARATION

I declare that this thesis entitled "Oxidation of Phenol using Silica Nanoparticles from Rice Husk Ash: Effect of Solvent" is the results of my own research except as cited in the reference.

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ABSTRACT

The oxidation of phenol using silica nanoparticles derived from rice husk ash (RHA) was investigated the effect of different solvents. In this research, RHA and phenol were aims to address on the challenges with an environmentally friendly approach towards pollutant removal and making production product. The silica nanoparticles were extracted it from rice husk until burning it to become rice husk ash and characterized catalyst produced by using technique Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), Power X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM). This research also to study the catalytic activity of silica on the oxidation of phenol by using various solvent by using the Gas chromatography–mass spectrometry (GCMS). The oxidation reactions were conducted under varying conditions, including different solvents such as acetone, methanol and toluene. The results revealed that the choice of solvent significantly influenced the oxidation efficiency of phenol, with acetone and methanol demonstrating superior performance compared to toluene. Additionally, the effects of solvent polarity, dielectric constant, and hydrogen bonding interactions on the oxidation kinetics were investigated. Overall, this study provides valuable insights into the role of solvent in influencing the catalytic activity of silica nanoparticles derived from RHA for the oxidation of phenol, contributing to the development of sustainable and efficient methods for making production product and environmental remediation. The findings underscore the importance of solvent selection in optimizing the performance of heterogeneous catalysts and highlight the potential of silica nanoparticles from RHA as promising catalysts for various oxidation reactions in the aqueous environments.

Keywords: Rice Husk Ash, phenol, silica nanoparticles, catalyst, solvent

Pengoksidaan Fenol menggunakan Nanopartikel Silika daripada

Beras Sekam Bakar: Kesan Pelarut

ABSTRAK

Pengoksidaan fenol menggunakan nanozarah silika yang diperoleh daripada beras sekam bakar (RHA) telah disiasat kesan pelarut yang berbeza. Dalam penyelidikan ini, RHA dan fenol bertujuan untuk menangani cabaran dengan pendekatan mesra alam terhadap penyingkiran bahan pencemar dan membuat produk pengeluaran. Nanopartikel silika diekstrak daripada beras sekam sehingga dibakar menjadi beras sekam bakar dan pemangkin berciri yang dihasilkan dengan menggunakan teknik Thermogravemetri Analyzer (TGA), Spektroskopi Inframerah Transformasi (FTIR), X-ray Diffractometer (XRD), dan Mikroskop elektron pengimbas (SEM). Penyelidikan ini juga untuk mengkaji aktiviti pemangkin silika terhadap pengoksidaan fenol dengan menggunakan pelbagai pelarut dengan menggunakan Kromatografi Gas–Spektrometri Jisim (GCMS). Tindak balas pengoksidaan dijalankan dalam keadaan yang berbeza-beza, termasuk pelarut yang berbeza seperti aseton, metanol dan toluena. Keputusan menunjukkan bahawa pilihan pelarut secara signifikan mempengaruhi kecekapan pengoksidaan fenol, dengan aseton dan metanol menunjukkan prestasi unggul berbanding toluena. Selain itu, kesan kekutuban pelarut, pemalar dielektrik, dan interaksi ikatan hidrogen pada kinetik pengoksidaan telah disiasat. Secara keseluruhannya, kajian ini memberikan pandangan berharga tentang peranan pelarut dalam mempengaruhi aktiviti pemangkin nanozarah silika yang diperoleh daripada RHA untuk pengoksidaan fenol, menyumbang kepada pembangunan kaedah yang mampan dan cekap untuk membuat produk pengeluaran dan pemulihan alam sekitar. Penemuan ini menggariskan kepentingan pemilihan pelarut dalam mengoptimumkan prestasi pemangkin heterogen dan menyerlahkan potensi nanozarah silika daripada RHA sebagai pemangkin yang menjanjikan untuk pelbagai tindak balas pengoksidaan dalam persekitaran akueus.

Kata kunci: Beras Sekam Bakar, fenol, nanopartikel silika, mangkin, pelarut

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Rice was the second most extensively eaten food and the primary staple for nearly 50% of the global population (Mithila, 2020). Rice was supplying more than 50% of the world's population with their basic grain (Sheaffer and Moncada, 2011). Rice husk (RH) or called as rice hulls is a byproduct produced when rice grains' outer shells and milling the process (Mithila, 2020). Rice husk was a significant by-product of the biomass and rice milling industries (Phonphuak, and Chindaprasirt, 2015). The tough outer layer of rice grains known as the "rice husk" shields the seed throughout the growth process (Kashif, and Fazul, 2017). Around 20% of the weight of the rice is made up of the husk, which is made of opaline silica and lignin (Syuhadah and Rohasliney, 2012).

Figure 1.1: Rice husk
(Source: Adam, 2023)

Rice husk ash (RHA) was a plentiful and sustainable agricultural byproduct produced during the milling of rice in the nations that produce rice. RHA has the greatest percentage of silica among all plant remains. The paddy plant produces 78% of the rice, 20% of the rice husk, and 2% of the waste products at a rice mill. Around 50% of the rice husk was made up of cellulose, 25% was lignin, and 20% was silica (Riza, and Rahman, 2015). RHA has includes around 90% silica, was lightweight, had a high specific surface area, and had a highly porous structure. Rice hulk ash has been used as an additive in a variety of products and materials, including refractory brick, the production of insulation, and flame retardant materials. Rice husk ash silica has different characteristics depending on the temperature and length of the fire process (Phonphuak, and Chindaprasirt, 2015). RHA was the end result of burning rice husk. When rice husk is burned, the majority of its evaporable components eventually disappear, leaving mostly silicate leftovers behind. While in certain locations it was field-burned as a local fuel, rice husk was sometimes used as fuel for parboiling paddy in rice mills. However, in some instances, the rice husks were only partially burned, which further adds to air pollution. (Singh, 2018).

In this study, silica was the term for inorganic ceramic materials made of silicon dioxide (SiO_2), which was the most prevalent substance on earth. (Yadav and Raizaday, 2016). The effectiveness of silica nanoparticles derived from rice husk for the removal of phenol from aqueous solutions. The study found that the silica nanoparticles had a high surface area and pore volume, which facilitated the adsorption and subsequent oxidation of phenol. The study also found that the use of the silica nanoparticles resulted in a higher rate of phenol removal compared to conventional oxidation methods (Fortuny et al., 1999). The use of silica nanoparticles derived from rice husk in combination with UV irradiation for the oxidation of phenol. The study found that the combination of silica nanoparticles and UV irradiation resulted in a higher rate of phenol degradation compared to using either method alone. The study also found that the use of the silica nanoparticles improved the stability and reusability of the catalyst. Silica has been listed as “generally recognized as safe” by the Food and Drug Administration (FDA). Due to this, silica-based nano- or microparticles have gained popularity in industry, the environment, and medicine (Seisenbaeva et al., 2021).

In scientific research, a solvent was a liquid or gas that dissolves or disperses a substance to form a solution. In the Latin word for the “solvent” is called as “solvō,” which means is “to loosen or solve.” The solvent was the component of a chemical solution that dissolves the solute and is present in the highest concentration (Anne, 2021). The choice of solvent can be an important consideration, as it can affect the solubility and stability of the compounds being studied. A nonpolar solute on wax that usually dissolves in a nonpolar solvent on xylene. Both polar and nonpolar compounds can frequently dissolve or cause the dissolution of molecules containing both nonpolar and polar components such as ethanol and acetone (Anne, 2021).

Solvent can be classified according to chemical bonds into three groups which are ionic liquids (molten salts with only ionic links), molecular liquids (molecules that melt), and atomic liquids (low-melting metals like liquid mercury or liquid sodium with metallic bonds) (Reichardt, 2002). The properties of the solvent, such as its boiling point, viscosity, and toxicity, should also be taken into account when selecting a solvent (Schoff, 2018). The ancient adage "similia similibus solvuntur" summarises how the categorization of solvents according to their chemical makeup allows for some qualitative predictions (Reichardt, 2002). Therefore, solvents serve an important purpose in chemistry, biology, pharmacology, and industrial uses (Anne, 2021).

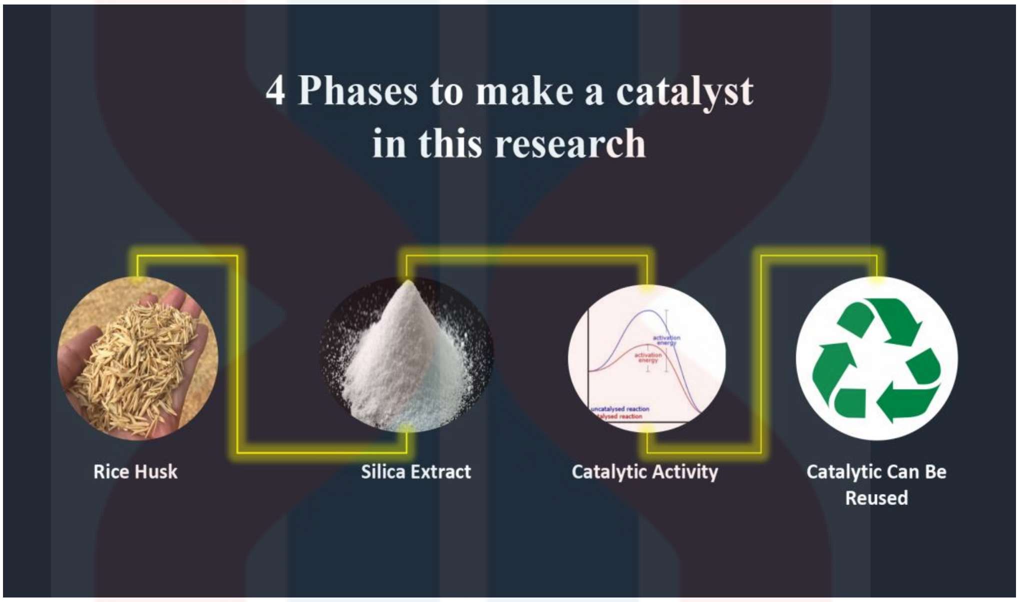

Figure 1.2: The flow of catalyst created and used in this research.

Figure 1.2 shows the flow of this research, which oxidation of phenol was used to extract the silica from RH. This catalyst will aid in accelerating the oxidation process' reaction rate, which will shorten the reaction's time and boost yield generation. Formula C_6H_5OH for the molecule of phenol is a dangerous and toxic pollutant, that must be decreased before being disposed of into the environment. It is typically found in waste water from industrial, agricultural, and home operations (Singh et al., 2020). The catalyst's objective is to create phenol byproducts with lower environmental impact, such as photographic chemicals, polymerization inhibitors, antioxidants, and flavouring agents (Adam et al, 2010).

1.2 Problem Statement

The RHA has was found to be a raw material that can be produced into value-added products with a multitude of applications while still being financially feasible. In addition to producing electricity, burning RH husk in a gasifier also solves the problem of waste management. If disposed of in an open area, the created RHA leads to environmental contamination and airborne infections. The RHA is really a possible source of amorphous silica used in high-value applications. Produced biochar may be used as fertiliser and is beneficial for enhancing the soil (Pode, 2015). However, burning the husks provided an even greater ecological issue when burying them left little place for the remainder of their crops to grow. The burning of the husks is the worst of the scenario for the local fauna since the fires spread rapidly, disrupt the ecology, and endanger everyone who lives nearby. (Lisa, 2019). The study aims to investigate the role of solvent properties, such as polarity and viscosity, on the catalytic activity and stability of the silica nanoparticles. Silica nanoparticles derived from rice husk have shown promise as an efficient and cost-effective catalyst for phenol oxidation. Selectivity is another essential characteristic of catalysts, which allows them to control reactions to produce more of the desired product and less of the undesirable byproducts.

Phenol was a toxic and persistent organic pollutant that was often present in industrial effluents and wastewater. Conventional treatment methods for phenol removal are not always efficient, and they can generate harmful byproducts. One promising approach to address this issue was the use of advanced oxidation processes (AOPs) that can mineralize phenol into harmless products. The phenol were the danger, teratogenicity, and mutagenicity, which have drawn much attention in recent years due to its high toxicity even at low doses. The production unit, desalter effluent, tank water drain, neutralised wasted caustic waste streams, and other sources can all contribute phenolic compounds to wastewater from a petroleum refinery. Hence, it was essential to treat these wastewaters effectively. Traditional wastewater treatment methods have a number of problems, such as insufficient or ineffective phenol removal (Sanaz et al., 2021). Phenol was a starting material used in the production of aspirin, explosives like picric acid, and polymers. Silver bromide crystals exposed to air are transformed into black metallic silver by the photographic developer's common phenol hydroquinone. Cresols (CAS 1319-77-3) were organic compounds on the three isomers of methylphenols, which are o-cresol, m-cresol, and p-cresol (Badanthadka and Mehendale, 2014). Wood preservatives like creosote include mixtures of phenols, particularly the cresols (Wade, 2018).

1.3 Objectives

In this research, there were two objectives which need to achieved. The objectives are:

1) To characterize the catalyst produced by using Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), Power X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM).

2) To study the catalytic activity of silica on the oxidation of phenol by using various solvent.

1.4 Scope of Study

In this scope of study, the discussion on the oxidation of phenol with help of catalyst produced. The catalyst will be a good support as indicated by the characterized to extracted the silica from rice husk. The first steps of this study will be started to extracting the silica from rice husk. Second steps of this study will be characterised and tested on the oxidation of phenol with a few parameters such as temperature, time, mass of catalyst, solvent and molaratio reacted hydrogen peroxide (H_2O_2). The result of the oxidation of phenol will identify the catalyst's condition and catalytic activity in order to ensure that they perform to their greatest potential when interacting in a process.

1.5 Significant of Study

The present study was extracting silica that was recovered from rice husk to create an organic catalyst. In order to avoid phenol from impairing water quality or the environment's ability to influence people. The catalyst that was created helps to speed up reactions and boost product yield. The rice husk may be completely utilised without harming the environment with the method provided. This significant of study was given more important to the human being and environment to keep the ecosystem to be stable and getting a good health with new research and technology.

CHAPTER 2

LITERATURE REVIEW

2.1 Rice Husk

RH was one of the oldest cultivated grains which the grass plant species name *Oryza sativa* (Sheaffer and Moncada, 2011). RH was the waste products left behind after the rice grains have been extracted, which mostly consist of silica (Nuamuah et al., 2012). Large amounts of rice husk are produced as a waste product during agricultural production of rice (Mithila, 2020). The world's largest production occurs in Asia, with the majority of the corp grown in China, India, Indonesia, and Thailand. (Sheaffer and Moncada, 2011). The amount of rice produced worldwide each year was 759.6 million tonnes (on a milled basis, 503.9 million tonnes). Hull makes up around 20% of the rice paddy, which results to an annual production of 152 million tonnes of RH waste (Mithila, 2020). Malaysia is one of the country to producing and production the rice. Rice is an important for Malaysian society due to its ability to promote agricultural activities and provide for a growing population (Fazleen and Stephan, 2017). As the pillar of Malaysian agricultural production, rice industry is an important to output and a large employer. Furthermore, Malaysians consume between 2.5 and 2.7 million tonnes of rice yearly, which it can making a staple meal and the main source of calories in the nation (Fazleen and Stephan, 2017). According to The Ministry of Agriculture and Food Industries (MAFI) has provided its assurance that, despite the myriad dangers facing its paddy and rice industries, including the effects of climate change. Malaysia's rice supply is still steady and sufficient to satisfy the demands of the people. Then, the percentage of RH generate increases automatically as rice production increases.

The main component of RH was cellulose, lignin, silica and moisture. The percentage of component in RH were cellulose (50%), lignin (25%–30%), silica (15%–20%), and moisture (10%–15%) (Singh, 2018). The elemental constitution (wt%) of rice husk is carbon in 41.92% , hydrogen in 6.34% , nitrogen in 1.85%, and sulfur in 0.47% (Hussein et al, 2022). This yearly renewable waste is made up of 70-72% in organic chemicals and 28-30% in inorganic compounds (Korotkova et al., 2016). RHA has a significant amount of silica (SiO_2). RHA has the capability to function as a highly reactive pozzolanic substance in controlled burning chambers (Zahid and Kazi, 2022). The silicates are the main byproducts of burning rice husk since the majority of its evaporable components are gradually lost. The makeup of the rice husks, burning temperature, and burning period all affect how the ash behaves (Singh, 2018). The typical particle size of rice husk ash is 3 to 10 micrometres. RHA that has been fully burned is grey to white in colour, while RHA that has been partially burned is blackish (Mahfooz et al., 2023). RHA has its own cellular structure prior to grinding, and the cellular surface of the ground RHA was dotted with pores. This is because RHA mostly consists of amorphous silica and only a little amount of crystalline phase, silica was responsible for the pozzolanic activity in mortar or concrete (Alengaram, 2022). The main inorganic component of rice husk was silica. It wass made using a 3N HCl acid pretreatment, burnt at 700°C for 4 hours, then subjected to leaching with 1.5, 2, and 3N NaOH. Amorphous silica particles with a large surface area make up the extracted findings (Ajeel et al., 2020).

RH has been used in numerous research fields up to this point, including tests for water treatment, as a replacement material in a cementitious matrix by making sustainable cement-based material, as a fuel in producing stream in parboiling process , as a source of silica in the production of ceramic, and upgrading the soil properties for agriculture by eliminating the heavy metal, reducing the soil bulk density, increasing the nutrient and manure.

RH will be using to resolves the waste disposal issue related to RH and results in the production of useful goods. It is presently a crucial catalyst for the chemical industry and a raw ingredient for the production of silicon. When compared to silica manufactured from quartz using the present technology, silica recovered from the RH is substantially better and more affordable. In comparison to the approach that includes fusing sand of a chosen certain grade, the process of extracting silica from RH consumes significantly less energy and is more energy-efficient.

2.2 Silica

In terms of chemistry, silica was a silicon oxide (SiO_2) (George et al., 2013). Silica is abundantly distributed in Earth's crust as silicate minerals and was also found in plants and cereals (Huang et al., 2022). Silica atoms were non-metal oxides and consist of four oxygen atoms surrounding one silicon atom in a etrahedral formation (Yadav and Raizaday, 2016). Silica was the foundation of 59% of the Earth's crust and more than 95% of all known rocks. As an inorganic material, silica nanoparticles possess uniform pore size, controllable particle size, large surface area, and easily modified surfaces owing to the presence of silanol groups (Si–OH), and excellent biocompatibility (Huang et al., 2022). Silica nanoparticles (SiO_2 NPs) are mesopores (2- to 50-nm pores) of silica that display unique physicochemical properties. These nanocarriers can be prepared in a variety of sizes and shapes including nanohelices, nanotubes, nanozigzags, and nanoribbons (Acharya et al., 2017). Silica nanoparticles are synthesized with surfactants like cetyltrimethylammonium bromide as templates (or structure-directing agents) and tetraethyl orthosilicate or sodium metasilicate (Na_2SiO_3) as the silica precursors (Acharya et al., 2017). Silica nanoparticles have been proposed for the controlled nanodelivery of silicon and other active ingredients to plants (Jianfeng et al., 2022). Silica is an excellent material for use in dentistry and other biological applications (George et al., 2013). Silicon oxida are the three major forms of this covalent combination, which quartz, tridymite, and cristobalite. This silicon atoms are all hexagonally tetrahedrally linked with oxygen (Mei et al., 2019).

Silica exists in many different phases, ranging from amorphous to crystalline. The distinct silica phases were briefly discussed and display various solubility behaviours. In the majority of terrestrial and aquatic settings, crystalline silica is a naturally occurring substance that persists (Southard, 2014). As was previously noted, quartz is the most common crystalline form of silica. Quartz was the most prevalent phase in nature and may be found in a range of forms, from enormous crystals to powders with an amorphous appearance. The three main forms of silica undergo transition, going from quartz to tridymite to cristobalite to vitreous (Iler, 1979). The amorphous state of silica can transform into the crystalline state at a certain temperature. The crystalline characteristic may generates when it was continuously heated and cooled over an extended period of time (Pandey et al., 2015). This is because differing temperatures affect how silica was formed, it was necessary to regulate the incineration's temperature. The ideal temperature was below 800 degree celsius or 973 Kelvin for the carbonization of RH in order to prevent the amorphous phase from becoming crystalline (Pandey et al., 2015).

The temperature requirements for the silica varied depending on its form. RHA may be used to create crystalline silica when rice husks are burnt at temperatures exceeding 850°C. It was possible to produce amorphous silica at temperatures lower than 700°C (Dhaneswara et al., 2020). Tridymite to cristobalite or cristobalite to tridymite required 1470°C, where as quarts to tridymite or tridymite to quartz needed roughly 870°C (Pandey et al., 2015). Then, the cristobalite's reversible reaction with vitreous required roughly 1700°C (Iler, 1979).

Amorphous silica must be extracted from the RH, which the temperature is the most important factor because temperature and burning duration may both affect how silica crystallises. Amorphous or noncrystalline silica was silicon dioxide without a crystalline structure. Amorphous silica has several uses and can either be naturally occurring or artificial (Christina, 2018). Amorphous silica was not a crystalline structure, where the arrangement of the crystals is random. The comparison between amorphous silica and crystalline silica has shown in Table 2.1.

Table 2.1: Comparison between amorphous silica and crystalline silica.

Characteristics	Amorphous silica	Crystalline silica
Structure arrangement	Does not have a well-defined crystal structure and randomly arranged in a disordered manner with the silicon and oxygen.	Do have well-defined crystal structure with ordered arrangements of silicon and oxygen atoms.
Properties	Transparent or translucent and has a glassy or non-crystalline appearance.	Opaque and has a crystalline appearance.
Density	Low density.	Higher density.
Formation	Produced through various methods, such as the precipitation of dissolved silica or the high-temperature treatment of silicon compounds.	Formed through natural geological processes, such as the cooling and solidification of molten rock or the gradual deposition of silica-rich sediments.
Used in	As a filler in various materials, including rubber, plastics, paints, and coatings. It improves mechanical properties, increases viscosity, and enhances durability.	Particularly quartz, is a primary component in glass manufacturing. It provides strength, transparency, and thermal resistance to glass products, including windows, bottles, and laboratory equipment.

The use of porous silica materials as catalyst supports has expanded because of their good chemical stability and the capacity to include different nanomaterials (catalysts) (Shide et al,, 2021). The properties of silica are essential to its functions, each of which necessitates a specific collection of properties. Among the several kinds of mesoporous silica materials discussed above, MCM-50, SBA-11, and SBA-12 are known to be effective adsorbent and catalytic supports (Shide et al,, 2021). As for the catalyst, it has to have amorphous silica, a high porous structure, a large surface area, and other characteristics that can encourage a high catalytic activity (Zhang et al., 2012).

2.2.1 Silica extraction from Rice Husk

The different of species have distinctive traits and components that can call for the employment of a different, more effective technique to extract silica. Numerous research have so far evaluated the effectiveness of the silica extraction approach on RH. Several methods, including the sol gel approach, the precipitation method, and the heat treatment method, are often employed. According to the majority of these research, sol-gel silica extraction produced the best results. Due to its capacity to regulate particle size, size distribution, and shape through systematic monitoring of reaction conditions, the sol-gel technique is frequently employed to create clean silica particles (Rahman and Padavettan, 2012). They have determined that the sol-gel method is superior to impregnation and coprecipitation for the preparation of catalysts because it yields structures with a higher surface area, more pores, a narrower size distribution, more homogeneity, and a significant number of tiny particles in loose structures. A catalytic membrane has been produced using the sol-gel technique. The construction of the catalytic membrane was examined using photocorrelation spectroscopy, nitrogen adsorption and gas permeation measurement (Zhou et al., 1998). The comparison of sol-gel, coprecipitation, and impregnation thas has been reported by Yao et al., 2018 on a Ni/Al catalyst is shown in the table 2.2.

Table 2.2: The comparison of methods for silica extraction.

	Sol-gel	Co-precipitation	Impregnation
Structure arrangement	The structure has a small particles in porous, loose structures with more uniform and more porous structures.	The structure has tight construction and flat surface.	The structure has irregular.
Pore volume	0.915 ml/g	0.404 ml/g	0.387 ml/g
Surface area	305.21 m^2/g	192.24 m^2/g	146.41 m^2/g
Ni loading wt.% (ICP-OES test)	8.50	8.04	10.5
Standard deviation (ICP-OES test)	0.84	1.00	1.30
XRD pattern	19.69 nm	26.17 nm	52.28 nm

Treatments are required to lessen contamination in order to get pure silica or high purity silica, and they can also enhance RHA's characteristics. The purity of the RH is improved by treatment, enabling a high silica concentration to be recovered from the RH. After the treatment of mineral acid leaching, >99% of silica was produced by RH burning at 600°C in inert atmosphere (Amutha et al., 2010). The structure of the rice husk will change as a result of all processes used, whilst the sample will alter in different ways depending on the technique used.

2.3 Catalyst, catalysis and its preparations

Three forms of catalysis which were enzymatic, homogeneous, and heterogeneous that are often applied in chemical reactions of catalysis processes. Catalysis was a word used to describe a process in which the presence of a material (the catalyst) that is not consumed during the reaction and must be removed later if it is not to be an impurity in the finished product influences the rate and result of the reaction (Andrei, 2007). Enzymatic was the biological catalysts (known as biocatalysts) that accelerate biochemical processes in living things (Robinson, 2015). Next, homogeneous catalysis is a categorised as either single-species or complex catalysis, however the line between the two is not always apparent (Helfferich, 2001). Then, heterogeneous catalysis is the design of technological systems must take interfacial interactions into consideration in significant application areas such as electrochemical energy conversion storage, corrosion, and tribology (Munz et al., 2023).

Catalysts may be created in a variety of methods, and each method will result in a unique set of characteristics, such as the catalyst's surface properties, its active components, and its adsorption sites. Common methods for creating catalysts include sol-gel, impregnation, and coprecipitation (Xia et al., 2011). Sol-gel, coprecipitation, and impregnation were employed as three distinct sorts of approaches to discover which strategy will provide the best catalyst. When compared to co-precipitation or impregnation techniques, the sol-gel process yields catalysts with the highest surface areas, more porous structures with narrow size distributions, more uniform structures, and better catalytic activity (Yao et al., 2018).

2.4 Phenol

Phenol (C_6H_5OH) was marked with a single 13C atom, it can take the form of any of four identically massed isomers, or a combination of them (Morton, 2010). It was a colourless, crystalline, somewhat organic solvent at room temperature having hydroscopic properties in water. Xylenols, oils, plasticizers, drugs, aspirin, detergents, oil refining, medicinal antiseptics, explosive colours, dye synthesis, the reagent in chemical analysis, and preservatives for leather and wood are just a few examples of the numerous chemical products that employ phenol (Afilah et al., 2014). Phenols are substances with an aromatic carbocyclic nucleus and a straight hydroxyl group connected to it. The common term for monohydroxybenzene was phenol (Smith et al., 1969). Since phenol was one of the most dangerous compounds, frequent usage of it may result in higher phenol levels being released into the environment, which will have an impact on ecosystem quality and interfere with nutrient cycling and biological ecosystems (Afilah et al., 2014).

CHAPTER 3

MATERIALS AND METHOD

3.1 Material

In this study, the material that used is the rice husk. The chemicals utilised to prepare the catalyst, cure the rice husk, and catalyse the research's catalytic reaction. The chemical that used in the research which were sodium hydroxide solution (NaOH), hydrochloric acid solution (HCI), toluene, phenol, acetone, and methanol.

3.2 Method

3.2.1 Extraction of silica from Rice Husk

The sol-gel method will be proceed on the silica extraction process from the RH were prepared the RH as a raw material. Then, every steps will used to proceed the extraction of silica process from RH.

The first steps is removing containment of RH. The rice husk was placed in a container with tap water and let to soak for some time. The rice husk sample that was required was found in the bottom of the container. The filth, including soil and dust, was then repeatedly washed away with tap water until the rice husk was finally rinsed with distilled water. After that, the sample dried at room temperature.

The second steps is the treatment in RH. A sample of 30.0g (RH) was weighed, and 24 hours were spent stirring it with on 500ml of 1.0 M of hydrochloric acid (HCl). After being thoroughly cleaned with distilled water until its pH was consistent (about 5.00). To get white rice husk ash (RHA), the sample was dried in an oven for

24 hours at 100°C and then burnt in a muffle furnace for 6 hours at 800°C with a heating rate of 10°C/min.

The third steps was the preparation of sodium silicate solution. The preparation of sodium silicate solution, which are 3.0 g of RHA mix was combined with 500ml of 1M of sodium hydroxide (NaOH) in a plastic container, and the mixture was constantly agitated for 1 hour. It was subsequently cooled to room temperature. After that, the mixture will filtered by using filter paper to produce a clear liquid solution called sodium silicate solution that was utilised to create the catalyst RHA.

3.2.2 The characteristic of the catalyst

Thermogravimetric analysis (TGA) with Differential Scanning Calorimetry (DSC), Fourier Transform Infrared Spectroscopy (FTIR), Powder X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM) with Energy-dispersive X-ray (EDX) were performed on the catalyst after it was manufactured.

Firstly, the technique used for the characteristics of the catalyst was thermogravimetric analysis (TGA). TGA was a thermal analysis technique used to study changes in the weight of a sample as a function of temperature or time under controlled conditions. TGA can determine the thermal stability of materials by observing weight loss or gain as a function of temperature. TGA can identify the composition of complex materials by analyzing weight loss patterns. Different components of a mixture may decompose or volatilize at different temperatures, allowing for their identification and quantification. Under TGA analysis was conjunction together with Differential Scanning Calorimetry (DSC) to getting a another result in the same analyzer. DSC was a thermal analysis technique used to measure the heat flow associated with physical and chemical changes in materials as a function of temperature. DSC was commonly used to study phase transitions such as melting, crystallization, glass transitions, and polymorphic transitions in materials. DSC also provides information about the thermal stability of materials by detecting decomposition or degradation events as temperature increases.

Secondly, the technique used for the characteristics of the catalyst was Fourier Transform Infrared Spectroscopy (FTIR). FTIR was a widely used analytical technique that provides information about the chemical composition, molecular structure, and bonding of materials. FTIR on Infrared light was used in this test to get information on the chemical interactions between the various components. The purpose of FTIR was to pinpoint the sample's RHA for chemical functional groups. The 4000-500 cm-1 scanning range was employed in this study.

Thirdly, the technique used for the characteristics of the catalyst was Power X-ray diffraction (XRD). XRD was a technique used in materials science and crystallography to determine the atomic and molecular structure of a crystalline material. XRD was utilised to gather information on the phase purity, crystallinity level, crystallite size, and unit cell characteristics. A beam was pointed towards the sample during XRD at an exact angle of incidence. The X-Rays deflect or diffract differently depending on the sample's crystal structure which is inter-atomic distances.

Finally, the technique used for the characteristics of the catalyst was Scanning Electron Microscopy (SEM). SEM was a powerful imaging technique used to visualize the surface morphology, topography, and composition of materials at high magnifications and resolutions. SEM was used to examine crazing patterns on the component's surface on higher magnification optical microscopy. The very porous catalyst structure of the sample was visible on its surface thanks to SEM, which also confirmed that the sample's amorphous condition lacked any crystal structure. Under scanning electron microscopy, a finely ground sample was examined to clearly show the material's porous character at low and high magnification. Energy dispersive X-ray analysis (EDX) was an analytical technique used in conjunction with scanning electron microscopy (SEM) to provide elemental analysis of materials. EDX was used to evaluate the sample's elemental composition. On conductive adhesive tape that was fastened to a stub of SEM, the sample powder was spread as thinly as feasible. It was possible to determine the sample's crazing patterns and the elemental analyses' average value.

3.2.3 The catalytic activity reaction in oxidation of phenol

In the oxidation of phenol will generated catalyst's activity to evaluated. A double-necked, round-bottom flask having a capacity of 250 ml and a water-cooled condenser. A typical run involved dissolving 20 mmol of phenol in 20 ml of acetone. The catalyst was activated at 100°C for 24 hours to eliminate any water molecules that had been adsorbing, and it was then cooled in a desiccator to reduce moisture content. The mixture was put into a flask with a round bottom and 0.1g of active catalyst, which was placed in an oil bath with a temperature control set at 75^{o}C.

Hydrogen peroxide (H_2O_2), 20mmol, was added dropwise to the rapidly stirred (600rpm) reaction mixture after the reaction temperature stabilised. The response took place for 4 hours. Each hour, an aliquot of 0.5 ml was taken out and filtered using a syringe. Prior to GC-MS analysis, 20μL of acetophenone was added to the sample as an internal standard. A few factors were then used to control the catalytic process, including the solvent, time, temperature, mass of the catalyst, the molar ratio of phenol to the catalyst's capacity.

Gas Chromatography Mass Spectrometry (GC-MS) was a powerful analytical technique used to separate, identify, and quantify complex mixtures of compounds. GCMS was verified the identities of the various products. It combines the principles of gas chromatography (GC) and mass spectrometry (MS) to provide detailed information about the composition and structure of samples. By comparing the GC of the respective pure hydroquinone, the products were further verified. In the MS, the compounds were ionized by electron impact (EI) or chemical ionization (CI) techniques, resulting in the formation of charged fragments.

The oxidation of phenol will influence of reaction to different solvent. To determine which solvent produces the best catalytic results, a variety of solvents including toluene, acetone, and methanol were employed. The procedure 3.5.1 was followed, with constant ratios of 20:20 mmol, mass constant at 0.10 g, and temperature of 75°C for 3 hours. After the reaction was finished, filtering should be used to separate the reaction mixture from the catalyst. Average values were utilised in the data presentation, and each reaction was carried out in triplicate.

CHAPTER 4

RESULT AND DISSCUSSION

4.1 Characteristics of catalyst

The catalyst's characteristics were designed to obtain results from any testing equipment used to examine the catalyst in the sample. These characteristics were analyzed using four methods: Thermogravimetric Analysis (TGA) with Differential Scanning Calorimetry (DSC), Fourier Transform Infrared Spectroscopy (FTIR), Power X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray (EDX).

4.1.1 Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC)

TGA was complete on the RHA powder sample from which the weight of sample was 0.0026 gram by using the TGA/DSC 2 HT Mettler Toledo. The TGA process has been set at a heating rate of 10°C/ min from 40°C to 600°C in the pin-holed aluminium crucibles while nitrogen gas was purged in 20mL/min. The TGA was utilised to understand vaporisation, absorption, decomposition, adsorption, sublimation, desorption, oxidation, and reduction (Shabnam and Amit, 2023). The TGA thermogram of RHA was showed a reduction on the sample weight loss. Fexofenadine hydrochloride (FEX) was non-sedating second-generation H_1 antihistamine inhibits H_1 histamine receptors in a reversible and competitive manner.

FEX was obtained as a white to off-white crystalline powder (Kumar et al, 2019). The weight loss has begin process when the fexofenadine hydrochloride (FEX) starting to be melt at the temperature 43.74℃ which the result in 8.8029% mass loss. Before the temperature started to melt, there was no dehydration prior to the melting point. Generally, the TGA test result has showed a desorption or drying on TGA curves graph in Figure 4.1. This curve was exhibits mass loss region which is then followed by a constant line because certain processes like drying where volatile compounds get evaporated and desorption (Nabeel and Zahraa, 2021). Unlikely, the test result in Figure 4.2 showed the TGA curves on precipitated silica (PS) and silica aerogel (SA) in single stage decomposition which was demonstrated on inflection points, mass loss rates, and residual masses, contributes to a more nuanced interpretation of the results (Zulhelmi et al, 2018).

There were some factor that affecting the TGA result. The first factor affecting TGA result was a weight of sample. The TGA result in the weight of sample has showed different between Figure 4.1 and Figure 4.2 which was 2.6 mg and 10 mg. This was because the sample Figure 4.1 has small quantity that can easily burned in the beginning between the precipitated silica (PS) and silica aerogel (SA) sample in Figure 4.2 that has more quantity that can long to burned into the process melting in TGA. The second factor affecting TGA result was a temperatures and heating rate. This was because sample in Figure 4.1 has lower temperatures and faster heating rate that lead to more rapid desorption and can affect the shape and features of TGA curves except Figure 4.2 that the weight sample on precipitated silica (PS) and silica aerogel (SA) give more influence on temperature and heating rate. The result in Figure 4.1 should used get higher in maximum temperatures on 1200℃ to indicated that the greater the weight loss, the less the crystallinity of the RHA (Ramadhansyah et al, 2012). The final factor affecting TGA result was different material in sample used. This because sample in Figure 4.1 showed the RHA has no additional material or chemcial that used it but for sample in Figure 4.2 showed the RHA has mixed with unsaturated polyester (UP) resin on amorphous precipitated silica (PS) and silica aerogel (SA) in TGA result (Zulhelmi et al, 2018).

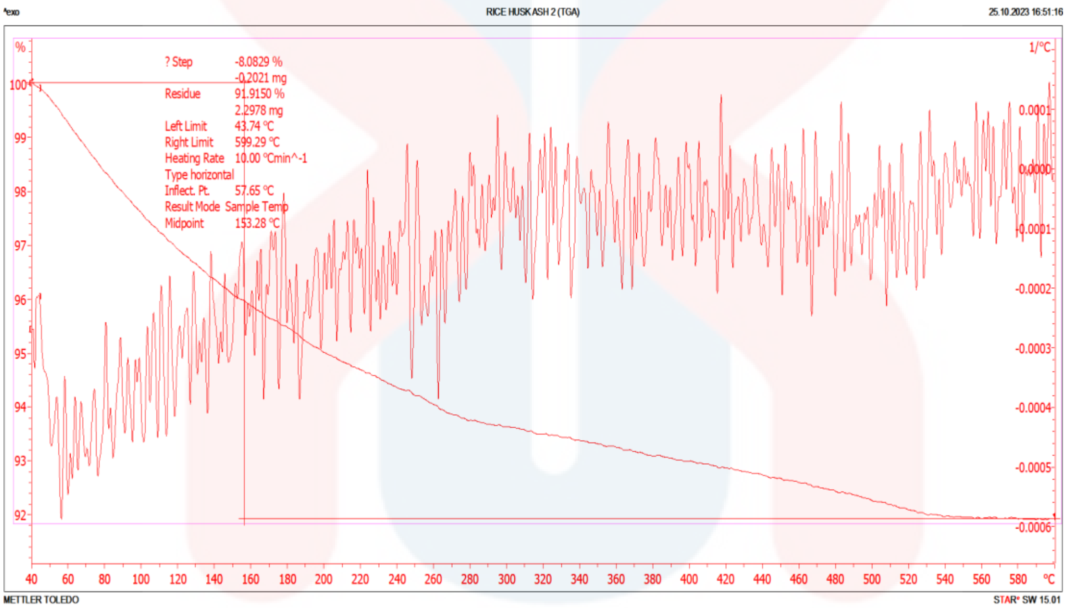

Figure 4.1: Thermogravimetric TGA curves treated by RHA

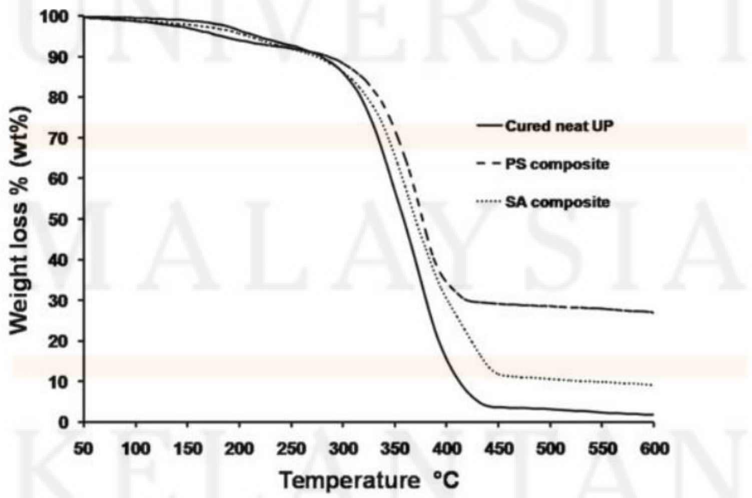

Figure 4.2: Thermogravimetric TGA curves by RHA on precipitated silica (PS) and silica aerogel (SA) (Zulhelmi et al, 2018)

DSC curves of RHA sample in Figure 4.3 showed a heat flow rate in 20mL/min and the temperatures range from 40℃ to 600℃. Exothermic curves was a major for the DSC curves as a test result. The exothermic was beginning the process when the heat flow rate increases above the baseline when heat was evolved by the sample. The shape of exothermic peak was a broader peak can suggest a more complex reaction pathway (Daud et al, 2020). DSC was usually presents two peaks: a small and broad peak at a lower temperature than a sharp and intense peak at a higher temperature (Adam West, 2018). The exothermic peak was start from 40℃ until 520℃ as a final peak that showed a some crystallisation temperatures due in the process. The data may be derived by determining the stiffness and glass transition temperature (Tg) of composites (Syifa' et al, 2020). The reaction on crystallisation showed nucleation on the initial formation of small crystalline domains within the material at the RHA (Russo et al, 2013). The transition from an amorphous to a crystalline state often involves an exothermic process. The endothermic also showed a heating rate was slower after finished the process at 520℃ to 600℃. The test result will be compared in Figure 4.4 which the exothermic peak at second peak was followed by rapid weight loss indicates the decomposition of cellulose, while third peak was ongoing decomposition of lignin and another organic constituent for continued to loss on the weight of rice husk that leached by HCl 3% (Dhaneswara et al, 2019).

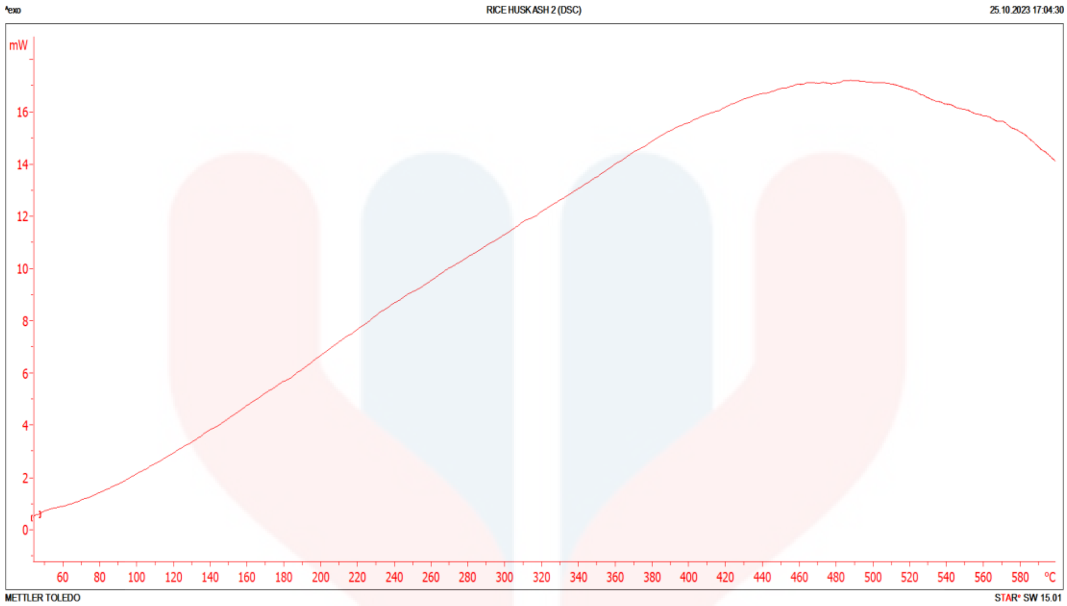

Figure 4.3: DSC analysis treated by RHA

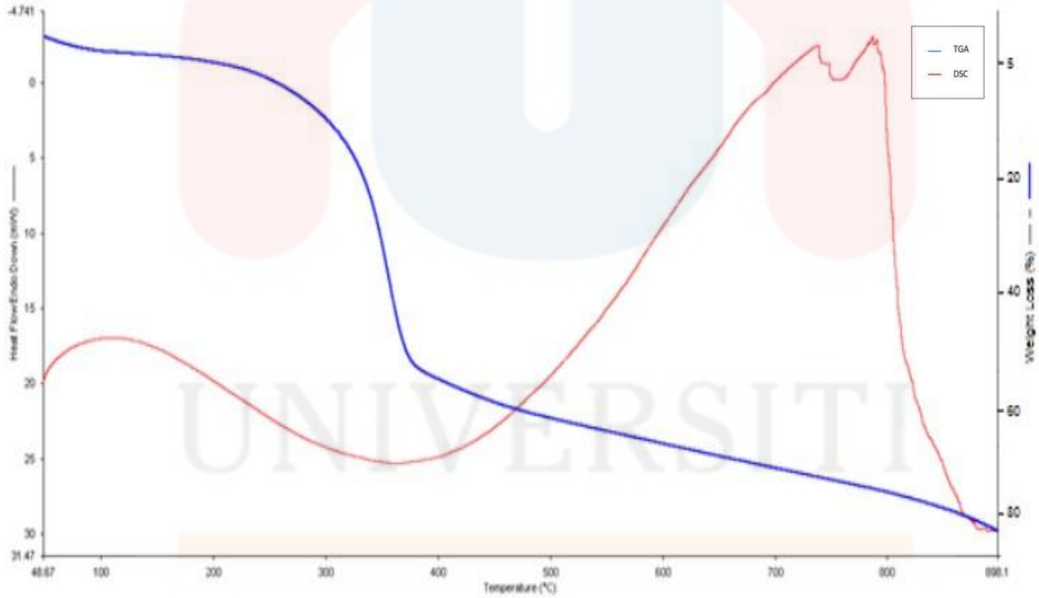

Figure 4.4: DSC and TGA of rice husk leached by HCl 3%. (Dhaneswara et al, 2019)

4.1.2 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy was used to analyse chemical changes on the surface of steam explosion-pretreated rice husk (Latika B and Dalip Kumar S, 2023) .Figure 4.5 demonstrates that the observed bandwidth between 3750.27cm^{-1} RHA was caused by adsorbed water molecules on the silica surface and isolated and surface functional O-H groups of (Si-OH) in the silica. Adsorbed water molecules were present between 3500 and 3000 cm, as shown by the absorption band centred at 3396.05cm^{-1} as reported by the N-H group. Rare-earth halides (RHAs) have a C-O wavenumber of 2360.61cm^{-1}. The performance peaked at 2113.01cm^{-1}, or in the range of 2500 and 2000cm^{-1}. Prior to 1500cm^{-1}, peaks were played at 1540.62cm^{-1}. The vibration of oxygen atom joined with the adjacent atoms in the asymmetric stretching vibrations of Si-OH band (Pallavi et al., 2012). The Si-O-Si bond's bending, symmetric, and asymmetric stretching vibrations are ascribed to the bands in Figure 4.5 spectra that are 1057.30cm^{-1} created C-O and 792.55cm^{-1} manufactured C=C. The chemically treated sample's silica functional group (Si-O-Si) seemed less intense than that of the raw rice husk because the base treatment removed inorganic elements from the rice husk surface (Samah et al., 2020).

Based the research on the FTIR test in Figure 4.6 showed a contained in RH that were rich in OH bonds besides C-H stretching vibrations, hemicellulosic sub-fractions and C-O-C stretching for glucose rings that can be determined at FTIR bands less than 3000cm-^{1}. The strong absorption peak at 1031.87 cm-^{1} indicates formation of Si-O-Si as an asymmetric and symmetric stretching band which was one of the obligatory compound that contain in rice husk (Fadilah et al., 2020). This demonstrates that rice husk was an effective source for producing high-grade amorphous bulk silica powder (Tzong-Horng Liou and Chun-Chen Yang, 2011).

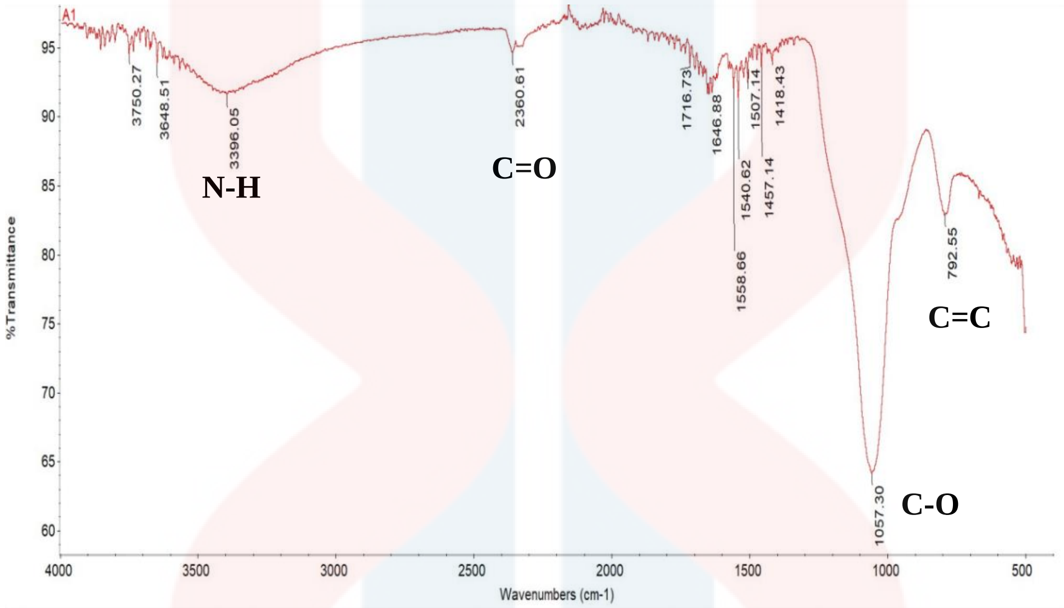

Figure 4.5: Extracted from FTIR spectroscopy of silica Rice Husk Ash

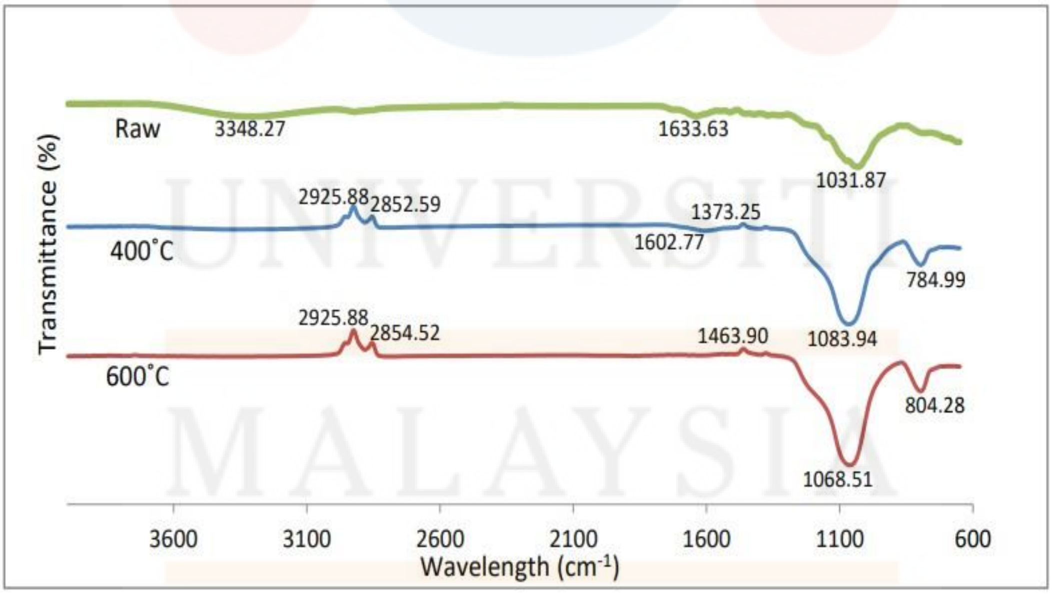

Figure 4.6: FTIR Spectra of SiO_2 obtained from Raw Rice Husk and via burning at 400°C and 600°C (Fadilah et al., 2020).

4.1.3 Power X-ray Diffraction (XRD)

The XRD equipment showed the x-ray diffraction pattern of silica formed by RHA. Researchers can determine the kinds and relative numbers of crystalline phases in a silica sample by comparing the recorded diffraction peaks to reference patterns for known crystal structures. XRD was a non-destructive method for analysing crystal structures in composite materials which that different crystal phases might result in varying material characteristics (Polini and Yang, 2017). XRD patterns can be used to gain a thorough understanding of crystalline structure, size, and orientation, dislocation density, phase identification, quantification, and transformation, lattice parameters, residual stress and strain, and thermal expansion coefficient of materials. Data may be used to tailor the material's properties for specific applications, such as increasing the mechanical strength of composites (Asif et al, 2022).

Silica powder was extracted from the resultant ash and treated with a NaOH solution before being titrated with HCl solution. XRD examination revealed that the removed silica powder was amorphous because silica becomes active in the amorphous state, it was preferred for the synthesis of silicon-based compounds (Kassim et al, 2022). In this context, the term "active" refers to the amorphous form of silica, which is more chemically reactive or readily engages in chemical processes than its crystalline version (Sldozian, 2014). This reactivity is generally linked to amorphous silica's chaotic and reactive surface structure.

The legitimacy of SiO_2 samples was validated by X-ray diffraction patterns produced from hydrochloric acid, as shown in Figure 4.7. There was no structure at all visible on the peak's huge, flat summit. The amorphous peak can be seen in the XRD pattern at 2θ-31°. The amorphous nature of silica was uncovered by the XRD pattern. According to the Table 4.1, the percentage of amorphous silica was approximately 43.4%, where as the percentage of crystalline silica was around 56.6%. Amorphous silica was created from SiO_2 tetrahedra that share a corner was the crystalline SiO_2 polymorphs on cristobalite and tridymite that have silica tetrahedra arranged in two or three layers (Gutiérrez-Castorena and Willam, 2018). Although an amorphous solid lacks the long-range organisation of a crystal, it does exhibit long-range interactions caused by chemical bonding or entanglements (Schmitz, 2017). Based on the XRD pattern of hydrochloric acid-treated silica RHA, we can infer that amorphous materials exhibit a broad hump and lack sharp diffraction peaks, while crystalline materials do.

As a result, amorphous silica had to be removed from RHA during the silica extraction procedure because amorphous silica was more dynamic than crystalline silica (Nzereogu et al. 2023). In non-distractive tests, amorphous silica surpasses crystalline silica and has superior mechanical properties which the crystalline silica performed better than amorphous silica to put it another way.

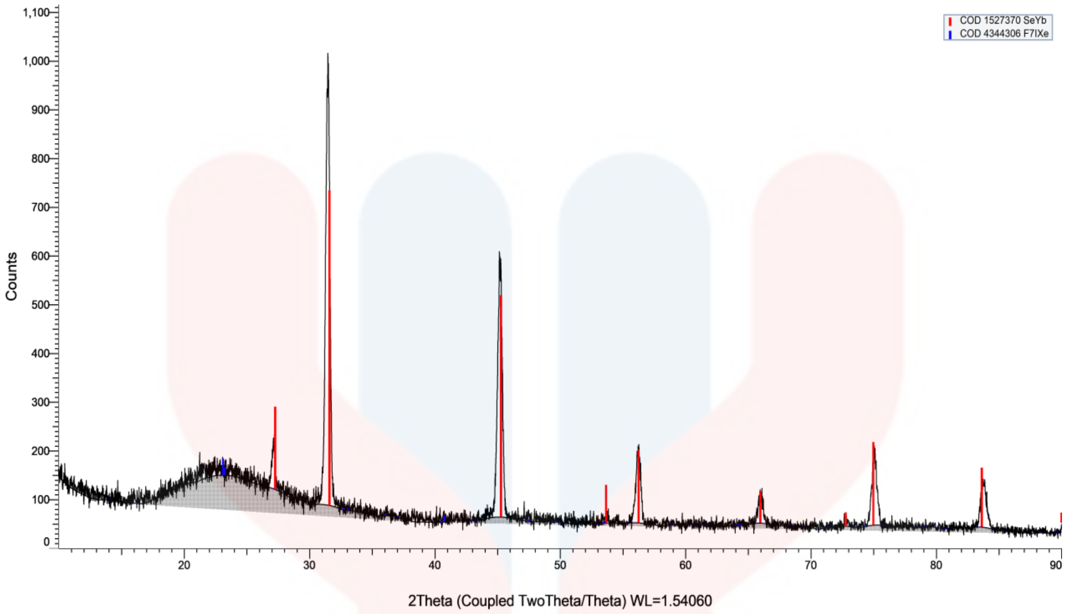

Figure 4.7: The Power X-ray Diffraction pattern for RHA.

Table 4.1: The percentage of amorphous and crystalline of silica from RHA.

Amorphous	Crystalline
43.4	56.6

4.1.4 Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX)

Scanning electron microscopy (SEM) has been used to examine the agglomerate microscopy of the RHA functionalized with RHA. The Figure 4.8 displays the SEM micrograph of RHA has showed that the under low of magnification on the RHA was topology consist of and some spherical surface particles. Its uneven morphologies and randomly arranged tiny particle sizes were shown on the RHA surface. Some smooth-surfaced particles were responsible for the low surface area and lack of porosity in RHA.

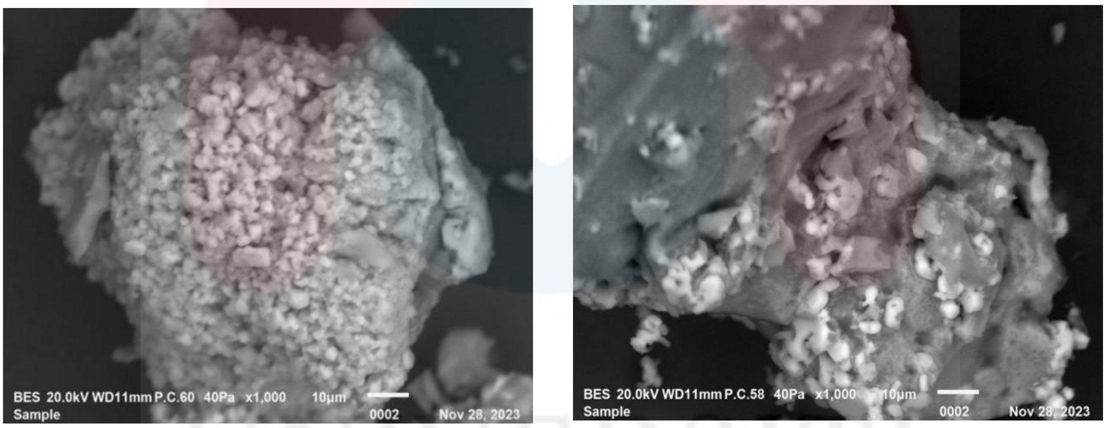

Figure 4.8: The SEM image of RHA.

Another research in Figure 4.9 was "Preparation and characterization of high-purity amorphous silica from rice husk" (Ngoc et al., 2018). Figures 4.8A and 4.8B show surface morphology (SEM images) of silica samples generated by alkaline extraction and SiO_2-precipitation, respectively. Silica products were main particle aggregates. Despite having larger primary particles (approximately 50 nm) than SiO_2-precipitated silica (about 25 nm), alkaline-extracted silica possesses gaps and holes (or a porous structure). Brunauer-Emmett-Teller (BET) has been confirm on SEM images at surface areas of 186.5792 and 68.2269 (m²/g) for alkaline-extracted and SiO_2-precipitated silica.

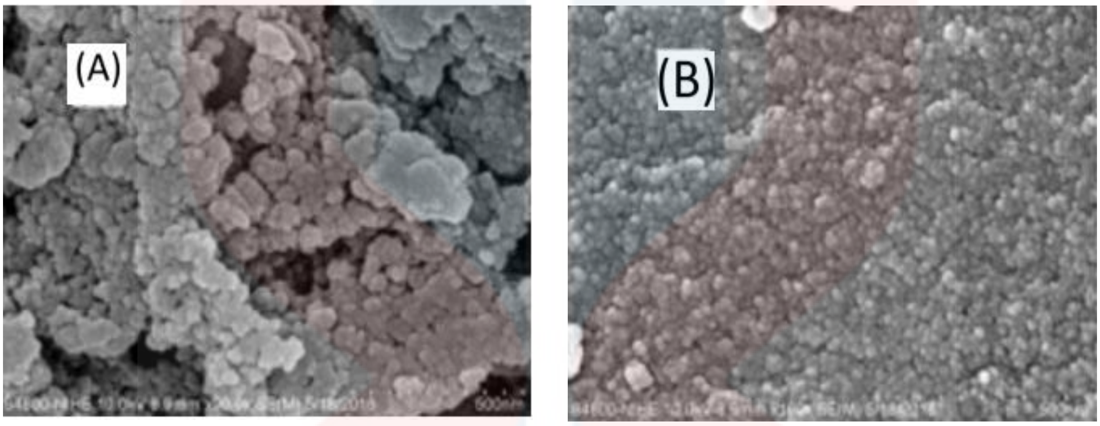

Figure 4.9: SEM images of silica produced from alkaline-extraction (A) and SiO_2-precipitation methods (B) (Ngoc et al., 2018).

An alternate technique of study includes the production of amorphous silica by alkaline extraction, reflux, and subsequent acidification with acetic or hydrochloric acid (Donanta et al., 2020). The surface morphology of amorphous silica was investigated using scanning electron microscopy images. The images were been captured at magnifications of 50,000× and 200,000×. Figure 4.10 A displays the grain of the rice husk-synthesized silica, which is uneven in form and measures 9-12 nm. Figure 4.10 B, which depicts a greater SEM magnification, clearly reveals grain sizes of different sizes, indicating that the synthesised silica is heterogeneous mesopore silica. The brightly coloured surfaces represent amorphous silica, whereas the darkly coloured surfaces show pore cavities.

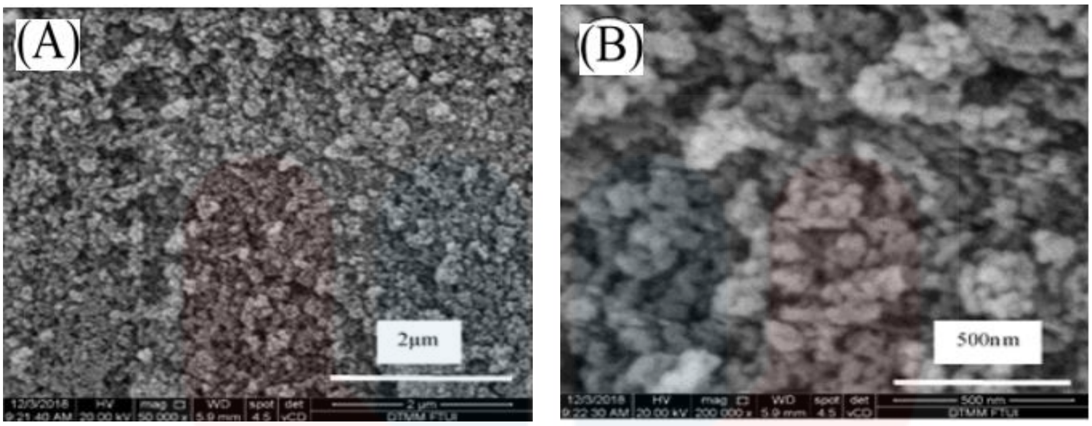

Figure 4.10: SEM micrograph of silica from rice husk using magnification:(A) 50.000×and (B) 200.000× (Donanta et al., 2020).

The energy dispersive X-ray analysis (EDX) at RHA elemental analysis at the has shows the different of spot in the SEM image displayed in Figure 4.11 and Figure 4.12. EDX microanalysis was an elemental analysis technique that generates distinctive X-rays in specimen atoms using incoming beam electrons on RHA (Manuel et al., 2018). The element content on RHA will been a main component was silicon (Si) or silica at spot 1 and spot 2. The sample of RHA at spot 1 and spot 2 has many different weight of element in Table 4.2. Table 4.2 has shows the percentage of element content in the sample of RHA at spot 2 was indicates a larger element weight percentage for Si (silicon) in 40.47%, while C1 (chlorine) was 36.54% and other element for O (oxygen) and Na (sodium) were 15.65% and 22.22%. Meanwhile, the percentage of element in RHA at spot 1 was shows a little element weight percentage for Si in 9.74%, while Na shows a larger element weight in 38.61% and other chemical for Cl, O and S (sulfur) were 37.90%, 13.61% and 0.14%. This element has shows that the silica on spot 2 was more larger weight than spot 1. This two spot has loss on ignition was caused by the removal of organic carbonaceous components such as cellulose, lignin, hemicellulose, D-galactose, proteins, and vitamins from rice husk (Datta and Halder, 2019).

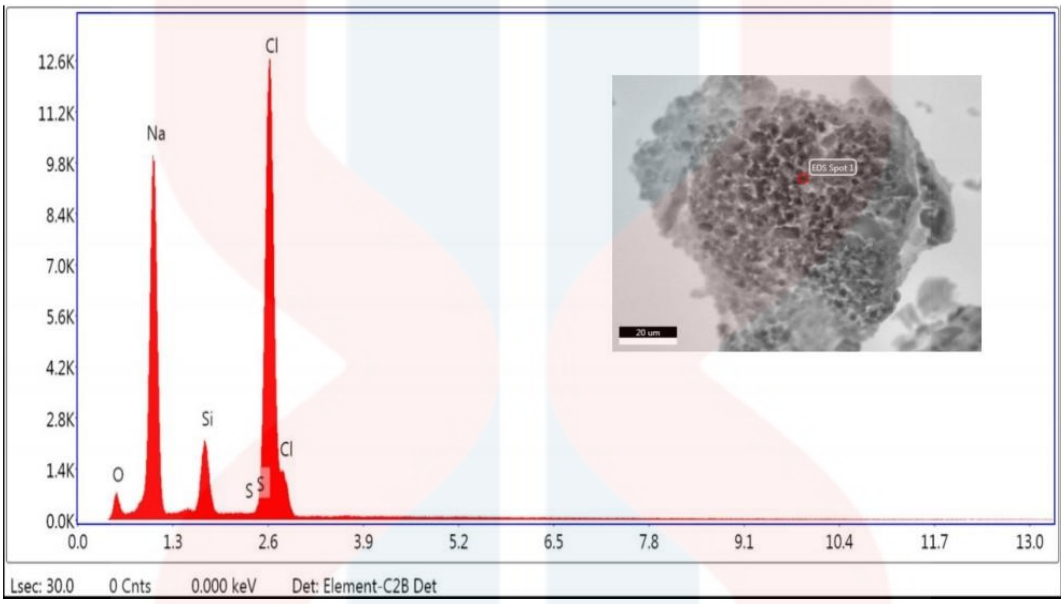

Figure 4.11: EDX profile of RHA with SEM micrograph at spot 1.

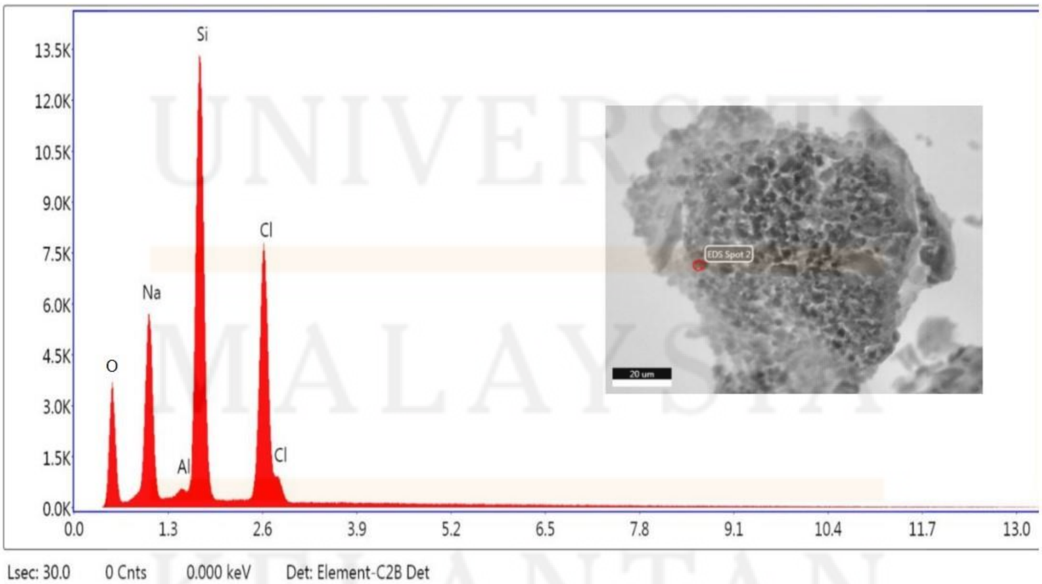

Figure 4.12: EDX profile of RHA with SEM micrograph at spot 2.

Table 4.2: Percentage of element content in the sample of RHA at spot 1 and spot 2.

Element	**RHA**	
	Spot 1 (weight %)	**Spot 2 (weight %)**
Sodium (Na)	38.61%	22.22%
Silicon (Si)	9.74%	40.47%
Chlorine (C1)	37.90%	36.54%
Oxygen (O)	13.61%	15.65%
Sulfur (S)	0.14%	0.00%

Based on the research in Figure 4.13 shows the EDX of nano rice husk ash (NRHA) has significant amount of oxygen and silicon. Other elements, including sodium (Na), magnesium (Mg), aluminium (Al), potassium (K), calcium (Ca), and iron (Fe), have relatively low percentages. There were no carbon peaks, indicating full combustion of RH (Vasamsetti et al., 2018). The EDX graph in Figure 4,9 also indicates that silica was the main element of Rice Husk Ash. The EDX spectra show that NRHA particles were stoichiometric. Stoichiometric was the molar connection of elements inside a chemical entity, such as a multielemental ion, molecule, or mineral (R. Hellmann, 1998). Based on EDX data in Table 4.3, the weight percentages of silicon and oxygen were found to be 57.63% for oxygen and 37.8% for silicon. Other element has calculated which were 0.53% in Na, 0.3% in Mg, 0.35% in Al, 0.9% in K, 1.49 in Ca and 0.93% in Fe. Thus, oxygen and silicon were the highest weight percentage of element in RHA.

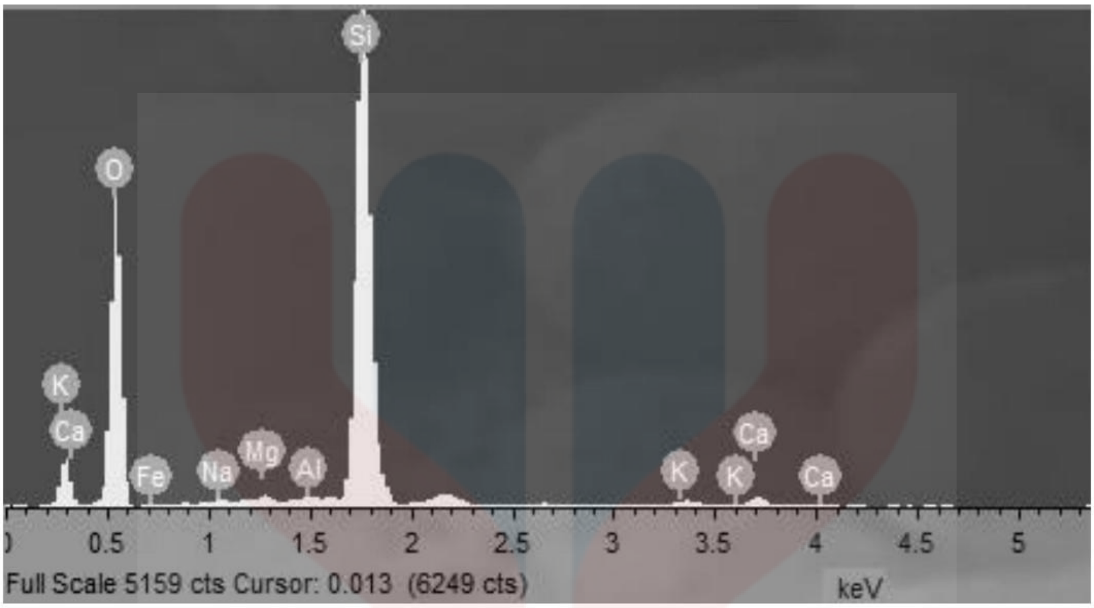

Figure 4.13: EDX profile of NRHA particles (Vasamsetti et al., 2018).

Table 4.3: Percentage of element content in NRHA.

Element	Weight%
Oxygen (O)	57.63%
Sidium (Na)	0.53%
Magnesium (Mg)	0.3%
Aluminium (Al)	0.35%
Silicon (Si)	37.8%
Potassium (K)	0.97%
Calsium (Ca)	1.49%
Iron (Fe)	0.93%

4.2 Catalytic activity of RHA in Oxidation of Phenol

In order to analyse the catalytic activity of the produced catalysts, phenol was oxidised using hydrogen peroxide as the oxidising agent and a number of parameters to evaluate the RHA. Studying the reaction's components in relation to how various solvents affect the reaction. Upon oxidation, the major molecule formed was phenol, 4-phenoxy- or known as hydroquinone monophenyl ether and acetic acid, phenyl easter, 4,4'-(1-methylethylidene)bis- occasionally showing up as a minor by-product.

The major molecule generated in Figure 4.14 was phenol, 4-phenoxy-, also known as hydroquinone monophenyl ether. Hydroquinone (HQ) was a dihydric phenol having two significant derivatives, namely monobenzyl and monomethyl ether of hydroquinone (Sarkar et al., 2013). It can be used as a photographic developing agent, dye intermediate, stabiliser in paints, varnishes, oils, and motor fuels. From the 1950s to 2001, hydroquinone was used in commercially accessible cosmetic skin lightening formulas in European Union nations, and from the 1960s, it has been marketed as a medicinal medication. It is also included in cosmetic formulas for coating fingernails and hair colouring (Francisco and Ana, 2013).

A minor by-product in Figure 4.15 that showed up was acetic acid, phenyl ester, also known as phenyl acetate. A phenyl ester was a chemical compound that consists of a phenyl group (a benzene ring) attached to an ester functional group. Phenyl esters were obtained in moderate to high yields by reaction of aliphatic and aromatic carboxylic acids with one equivalent of diphenyl carbonate in the presence of catalytic amounts of tertiary amine bases (Oliver and Micheal, 2015). Phenyl esters are widely utilised in the fragrance and flavour industries to impart distinct odours or tastes. It can enhance the aromatic and flavour qualities of perfumes, colognes, and food items (Arceli et al., 2003).

A minor by-product at toluene reaction in Figure 4.16 that occasionally showed up was phenol, 4,4'-(1-methylethylidene)bis- or known as Bisphenol A (BPA). BPA was one of the main chemical compounds used in a wide variety of everyday products due to its physicochemical characteristics (J. L. Torres-García et al., 2022). BPA was an organic synthetic substance utilised as a monomer in the production of polycarbonate plastic, which was widely used in food and beverage containers, medical equipment, thermal paper, and dental products (Ilaria et al., 2020).

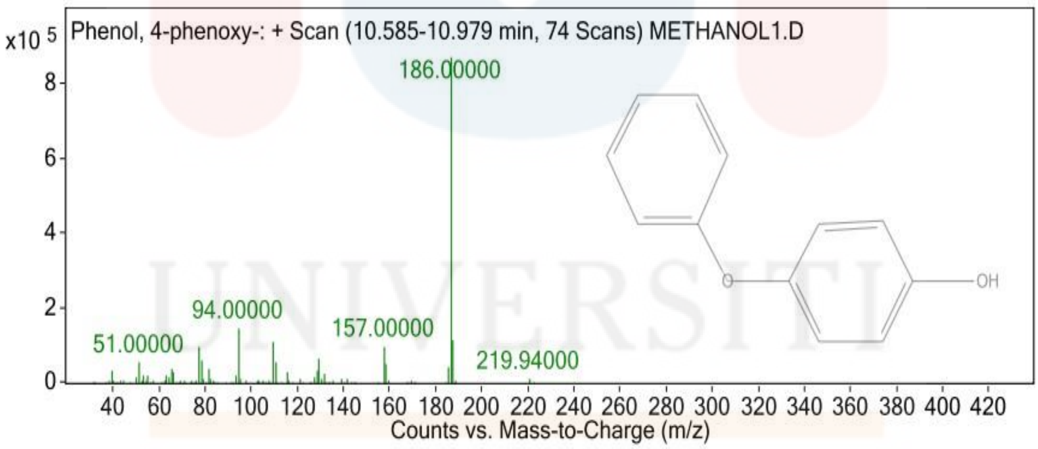

Figure 4.14: The major product in hydroquinone monophenyl ether.

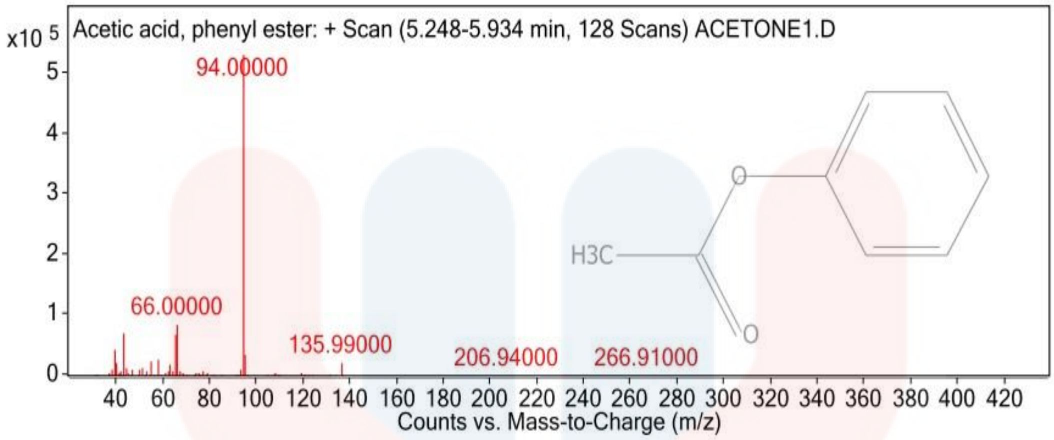

Figure 4.15: The minor by-product in phenyl acetate.

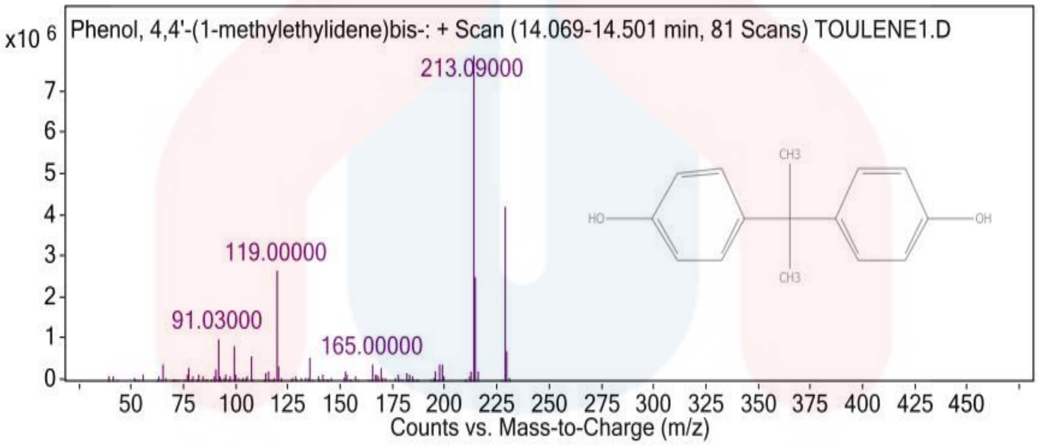

Figure 4.16: The minor by-product in Bisphenol A.

The tallest peak at m/z 94.1000 in Figure 4.17 was showed that the most abundant ion identified has a mass-to-charge ratio of 94.1000 for the solvent of acetone. This is the basic peak, which reflects an intact phenol molecule that was ionised without fragmentation. The peak at m/z 94.1000 can represent the molecular ion of phenol (C_6H_5OH), which has a molecular weight of 94.

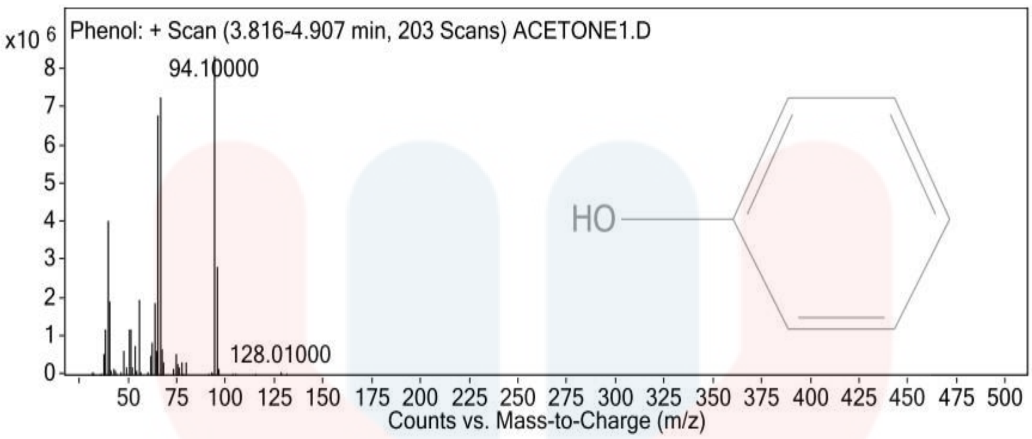

Figure 4.17: Mass-to-charge ratio (m/z) in the solvent acetone.

Figure 4.18 shows a peak at m/z 94 for the molecular ion of phenol (C_6H_5OH) in the solvent methanol. The peak at m/z 66 may reflect a fragment of an oxidation product or a smaller organic molecule generated during the oxidation process. This peak was m/z value corresponds to the molecular weight of phenol, which was 94 g/mol. The peak at m/z 128 was more difficult to assign without extra information. It might be a dimer or an oxidation product with a larger molecular weight than phenol.

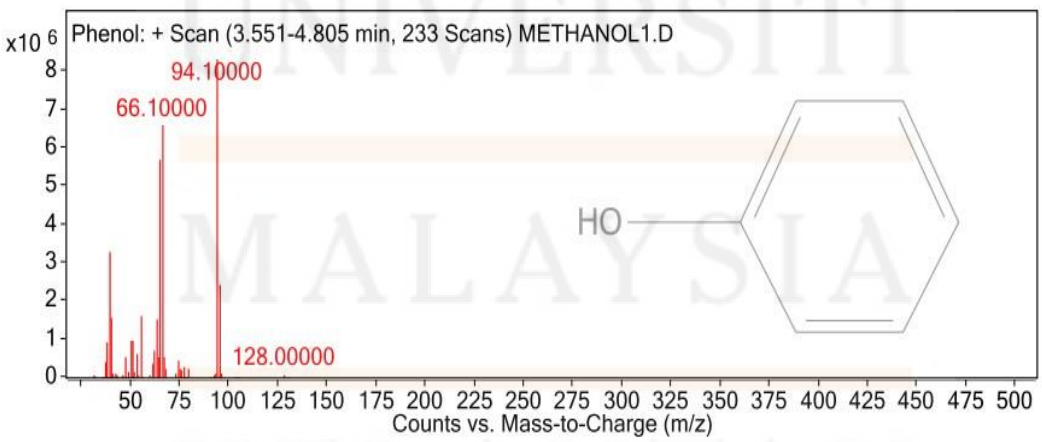

Figure 4.18: Mass-to-charge ratio (m/z) in the solvent methanol.

The peak at m/z 94 in Figure 4.19 might represent the molecular ion of phenol, which has a molecular weight of 94 g/mol in the solvent toluene. This shows that the sample may include some unreacted phenol. This was the basic peak, which shows an unbroken phenol molecule that was ionised without fragmentation. This graph most likely reflects the analysed substance's mass spectrum, exhibiting the distribution of ions according to their mass-to-charge ratio. Such information was critical for identifying the chemicals in the sample and establishing their molecular weights, providing significant insights into the composition and structure of the material study.

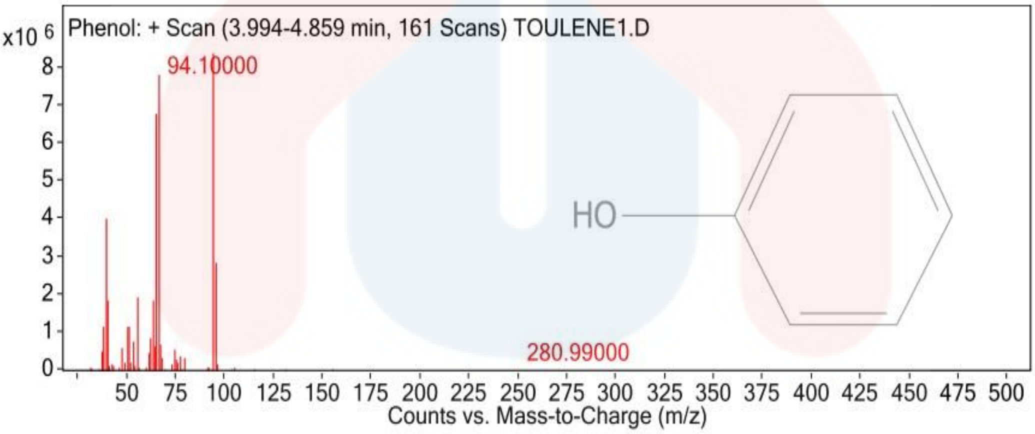

Figure 4.19: Mass-to-charge ratio (m/z) in the solvent toluene.

4.2.1 Effect of reaction to different solvent

Acetone, methanol and toluene were a some of the several solvents used in this investigation. The oxidation procedure included a 0.10 g catalyst that ran at 75 °C for three hours with a 1:1 molar ratio of phenol to hydrogen peroxide (H_2O_2). Only phenol and the internal standard (acetophenone), which was identified by the Gas chromatography–mass spectrometry (GC-MS) from the catalytic result for the acetone, methanol and toluene solvent, remained after three hours of operation. Figure 4.20 shows the conversion rate in acetone was around 38%, with 97% selectivity for hydroquinone monophenyl ether. Acetone was used as the steady-state solvent for phenol oxidation using a RHA catalyst.

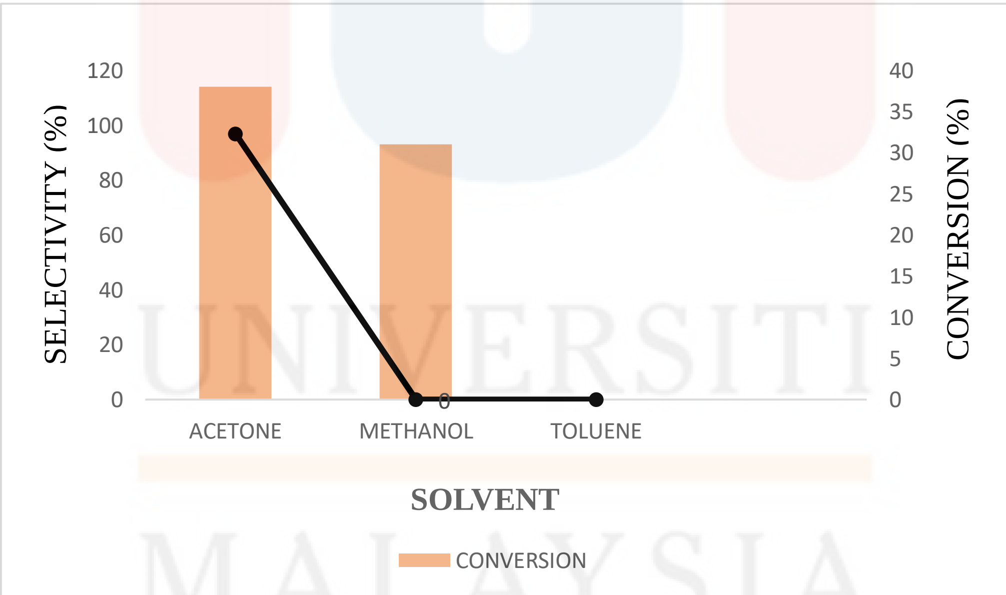

Figure 4.20: The percentage conversion and selectivity to hydroquinone at different reaction of solvent in the oxidation of phenol.

In summary of the effect on different of solvent, this study shows the best solvent for the reaction which were acetone and methanol challenges in achieving environmentally safe phenol removal. This solvent can be a good for human health on the product used by making cosmetics production, like skincare, perfume, nail polish and hair dye. Unfortunately, the reaction of solvent on toluene was not achieving environmentally safe phenol removal because the chemical was making plastic production and can be harmful on human health, like skin, brain and prostate gland of fetuses, infants and children. Thus, acetone and methanol was chosen as the steady-state solvent for phenol oxidation, with RHA functioning as the catalyst.

CHAPTER 5

CONCLUSION AND RECOMMEDATIONS

5.1 Conclusion

In conclusion, the oxidation of phenol using silica nanoparticles derived from rice husk ash represents a significant advancement in green chemistry. Silica nanoparticles, due to their high surface area and unique properties, serve as efficient catalysts in various chemical reactions. When applied in the oxidation of phenol, they offer promising results in terms of both reaction efficiency and environmental sustainability. Researchers were focused on meeting the growing demand for natural, organic, and ecologically friendly products. Heterogeneous catalysts have regenerative properties. This work creates a green catalyst using rice husk from agricultural waste as a source of silica. Amorphous silica was synthesised and burning the rice husk to produce RHA. Numerous research have explored heterogeneous catalysts made from rice husk silica. However, most catalysts use a non-renewable metal element to enhance their activity. RHA catalysts have several advantages, including low cost, environmental friendliness, and high efficiency. Further research and optimization efforts were warranted to enhance the efficiency and applicability of this catalytic system in real-world environmental remediation scenarios.

There a several analyses from TGA, DSC, XRD, SEM, EDX, and FTIR has been investigations were employed to characterise the development of RHA and its effective integration of rice husk silica to understand its shape. The TGA analysis on the drying curve was typically exhibits a slight weight loss at lower temperatures, corresponding to the removal of physically adsorbed or surface-bound moisture. This stage was characterized by a gradual decrease in weight as the sample undergoes dehydration. DSC analysis on exothermic processes may occur on the crystallization of amorphous phases or redox reactions involving inorganic constituents in the ash. These reactions contribute to the overall thermal behavior of the ash sample and can be characterized by distinct exothermic peaks in the DSC curve. The FTIR analysis, as demonstrated was successfully to provided proof for the functional group and chemical bond present in the samples' RHA. The peak in FTIR analysis indicates the functional group, degree of silanol condensation, and carbon content of the sample. The XRD diffractogram in RHA test has amorphous silica outperforms crystalline silica and has greater mechanical qualities, but crystalline silica fared better. In addition, the SEM picture shows the sample's surface structure before and after incorporating an organic component, including agglomeration, spherical, and irregular rock-like particles. EDX analysis has showed that the organic ligand was effectively immobilised in the silica matrix, as evidenced by the comparison of element percentages in the RHA catalyst before and after immobilisation.

A parameter on effect of solvent has been used to synthesis the RHA catalyst in oxidation of phenol using hydrogen peroxide as the oxidising agent. The reaction's main result in GCMS was hydroquinone monophenyl ether, which was soluble in acetone and methanol. This solvent can produced many product, especially cosmetics production. However, solvent in toluene has some dangerous chemical to produced the product and harm health to human. Thus, without catalyst, it cannot facilitate or accelerate chemical reactions in catalyst present.

5.2 Recommendations

This research created an organocatalyst by immobilising RHA silica. Chemicals used during catalyst production were crucial for developing efficient catalysts. Different solvents affect the catalyst's morphology, including structure, surface, pore structure, shape, and other variables. This may potentially influence the catalyst's active site. Therefore, the appropriate solvent setting was needed. The thesis' conclusion has been completed. Further study can be conducted as mentioned below:

- identified the RHA on different solvent by optimize the reaction conditions on solvent-to-reactant ratio to maximize phenol conversion and minimize undesirable by-products.
- Use the RHA catalyst for a variety of applications.
- investigate the possibility of combining multiple solvents or solvent mixtures to enhance the extraction efficiency.
- identify eco-friendly solvents that minimize environmental footprint and promote sustainable catalytic processes.

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APPENDIX A

Figure A: Rice husk dried in oven for 24 hours at 100°C

APPENDIX B

Figure B: Rice husk burnt in 400°C until 700°C to obtain rice husk ash

APPENDIX C

Figure C: Rice husk ash after burnt in muffle furnace for 800°C in 6 hours

APPENDIX D

Figure 4: White powder of silica rice husk ash