Oxidation of Phenol using Nanoparticles from Rice Husk Ash: Effect of Mass

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DECLARATION

I declare that this thesis entitled "Oxidation of Phenol using Silica Nanoparticles from Rice Husk Ash: Effect of Mass" 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 mass of catalyst. 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 mass by using the Gas chromatography–mass spectrometry (GCMS). The oxidation reactions were conducted under varying conditions, including different masses such as 0.10g, 0.20g and 0.30. The results revealed that the choice of different mass significantly influenced the oxidation efficiency of phenol, with weight 0.20g demonstrating superior performance compared to 0.10g and 0.03g. 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, mass of catalyst.

Pengoksidaan Fenol menggunakan Nanopartikel Silika daripada Beras Sekam Bakar: Kesan Jisim.

ABSTRAK

Pengoksidaan fenol menggunakan nanozarah silika yang diperoleh daripada abu sekam padi (RHA) telah disiasat kesan jisim mangkin 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 sekam padi sehingga dibakar menjadi abu sekam padi dan pemangkin berciri yang dihasilkan dengan menggunakan teknik Analisis Termogravimetrik (TGA), Spektroskopi Inframerah Transformasi Fourier (FTIR), Belauan Sinar-X Kuasa (XRD), dan Elektron Pengimbasan Mikroskopi (SEM). Penyelidikan ini juga untuk mengkaji aktiviti pemangkin silika terhadap pengoksidaan fenol dengan menggunakan pelbagai jisim dengan menggunakan Kromatografi Gas–spektrometri jisim (GCMS). Tindak balas pengoksidaan dijalankan dalam keadaan yang berbeza-beza, termasuk jisim yang berbeza seperti 0.10g, 0.20g dan 0.30. Keputusan menunjukkan bahawa pilihan jisim berbeza secara signifikan mempengaruhi kecekapan pengoksidaan fenol, dengan berat 0.20g menunjukkan prestasi unggul berbanding 0.10g dan 0.03g. 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: Abu Sekam Padi, fenol, nanozarah silika, mangkin, jisim mangkin.

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LIST OF ABBREVIATIONS

RH	Rice Husk	1
RHA	Rice Husk Ash	2
HCl	Hydrochloric Acid	18
NaOH	Sodium Hydroxide	19
SiO_2	Silicon Dioxide	3
H_2O_2	Hydrogen Peroxide	7
TGA	Thermogravimetric Analysis	19
DSC	Differential Scanning Calorimetry	19
XRD	X-Ray Diffraction	20
FTIR	Fourier Transform Infrared Spectroscopy	20
SEM	Scanning Electron Microscopy	21
EDX	Energy Dispersive X-ray Analysis	21
FEX	Fexofenadine Hydrochloride	24
GC-MS	Gas Chromatography Mass Spectrometry	22

LIST OF SYMBOLS

CHAPTER 1

INTRODUCTION

1.1 Background of Study

In Malaysia, paddy has developed into a significant crop that is used for the production of rice and rice husk, both of which are employed in a wide range of industries throughout the country. Figure 1.1 shows the milled rice production tonnage in Malaysia has been rising over the years (Daniel Tong Kuok Ho 2019). The process of dehusking rice results in rice husk (RH), a by-product that is often discarded. After burning rice husks, the residual components are 20% carbon dioxide, 80% air and 20% ash. Rice husk ash may be used to produce silica gels and particles at a reasonable cost. (RHA Silica). Silica accounts for approximately 60% of RHA, making it the most prevalent component. RH is one of the agricultural wastes that has gained popularity due to its several benefits, including low cost, absence of rivalry with food supply, reduced reliance on renewable energy, and the ability to reduce greenhouse gas emissions.

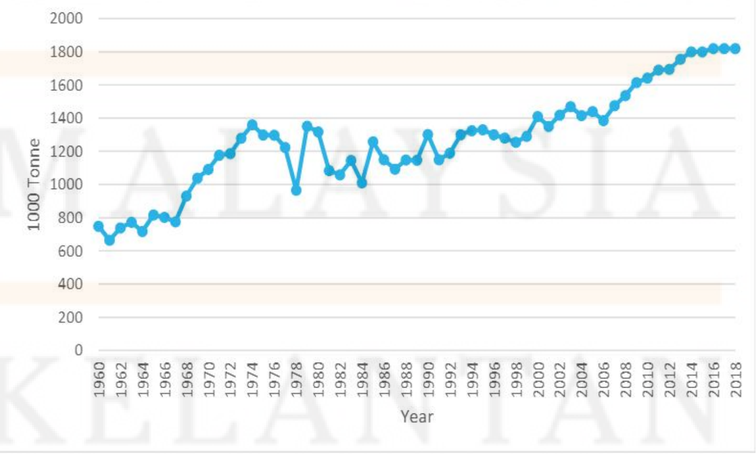

Figure 1.1: Production of Milled Rice in Malaysia

(Published by Daniel Tong Kuok Ho 2019)

Figure 1.2 shows that rice husk was a major by-product of the rice milling and agro-based biomass industry (Phonphuak, and Chindaprasirt, 2015). Rice grains have a tough outer coating called the "rice Husk" that shields the seed during the growth process (Kashif, and Fazul, 2017). The husk, composed of opaline silica and lignin, accounts for about 20% of the total weight of the paddy (Syuhadah and Rohasliney, 2012). Rice husk is a versatile agricultural byproduct that can be used for many different purposes. It can be burned for power, utilized as a raw material to create high-value goods, and included in food, bedding, and construction materials. A sustainable, circular economy that reduces waste, conserves resources, and lessens the effects of climate change may be fostered by using rice husk.

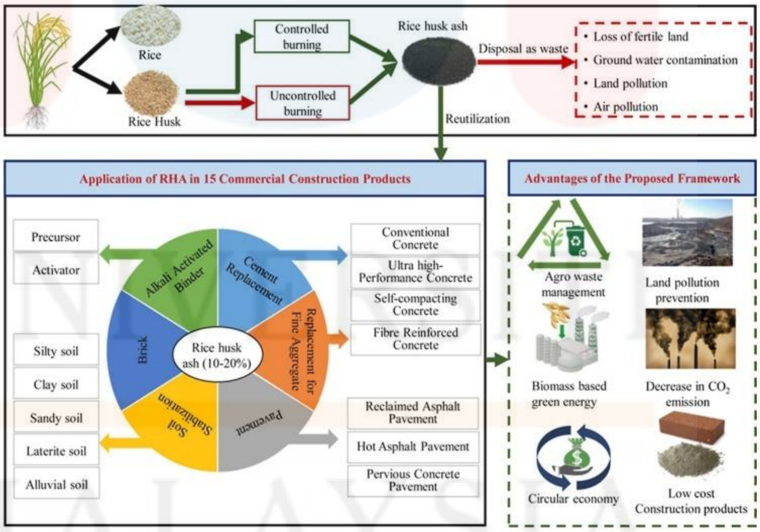

Figure 1.2: Rice husk was a major by-product of the rice milling and agro-based biomass industry.

(Phonphuak, and Chindaprasirt, 2015).

Figure 1.3: Rice husk

Published by Adam Duckett (2023)

The RH shown in Figure 1.3 has components that include cellulose, hemicellulose, lignin, and ash. Furthermore, this biomass waste has distinct features, such as a high silica content, which is an inorganic chemical component of ash. Rice husk ash (RHA) is an abundant and sustainable agricultural by-product created in rice-producing countries during the milling process. Its silica content is the greatest of any plant by-products. 78% of rice, 20% of rice husk, and two percent of rubbish are all by-products of the paddy plant at a rice mill. About 50% cellulose, 25% lignin, and 20% silica can be found in rice husks, as reported by (Rahman & Padavettan, 2012) . RHA is extremely porous, has a high specific surface area, is lightweight, and contains over 90% silica. Rice husk ash has been utilized as an addition in many different goods and processes, including the creation of insulating materials, refractory brick, and flame retardant materials. Rice husk ash silica's properties vary with burning temperature and time (Phonphuak and Chindaprasirt, 2015). When rice husk is burned, it produces RHA as a byproduct. Since most of the evaporable components of rice husk are lost over time during combustion, the silicates are the primary byproducts. Rice husk is sometimes used as fuel for parboiling paddy in rice mills, despite its traditional use as a local fuel (burned in the field). However, in these cases, only a portion of the rice husks were burned, which further contributes to air pollution (Singh, 2018).

The outer shell of the rice grain is hard and protective, encasing the edible portion within. The husk of rice is usually dry and fibrous, with a light brown colour. The rice milling business produces a valuable by-product known as rice husk. This versatile material has several practical applications. To produce steam, which in turn generates electricity, and to dry materials, it is commonly used as a fuel in boilers. Additionally, it may be utilised as a base for mushroom growing as a component in the manufacturing of biofuels, construction materials, and animal feed. It is inexpensive, readily available, and renewable, which are just a few of the numerous benefits of rice husk.

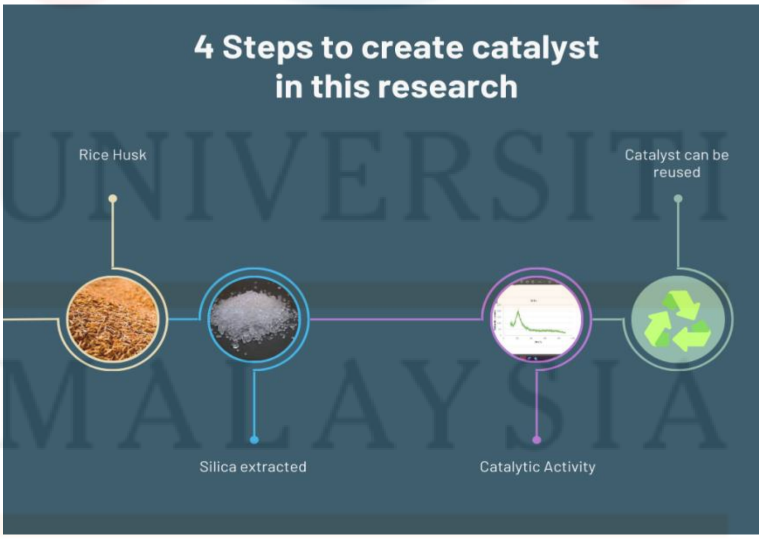

Figure 1.4: The flow of catalyst created and used in this research.

Figure 1.4 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

Numerous researchers use RH as a research material because of its high silica content in producing silica base material and discovering their essential or used. Although it is often burned in the field as a local fuel, it has found use in specific applications, such as parboiling paddy in rice mills. However, this results in incomplete RH combustion, which is a source of air pollution. Because of inefficient utilization, excess RH can also become a global problem. RH is often burned in the open, which has negative effects on both the environment and human health. The extremely low rate of RH waste usage can be attributed to the material's resistance to breakdown, low nutritional value, and high ash concentration. Currently (2015) (Alshatwi et al.).

Therefore, it is crucial to discover methods to convert RH into environmentally beneficial materials. Silica may be extracted from RH at a lower cost and with less side effects on the environment. Catalysts constructed from RH-recovered silica have been the subject of a large body of research, but only a small number of papers have investigated catalysts immobilized with organic chemicals more closely associated with biological activities. The catalyst's function is to accelerate the reaction and boost the yield. Gupta et al. (2019) showed that the fabricated SBA-16-G catalyst could generate a high yield of product with minimal effort.

Phenols are a class of chemicals that are both highly toxic and notoriously difficult to remove from the environment once they have been released. It is produced or supplied naturally through wastewater or effluent from human activities like farming, industry, and mining (Abd Gami et al., 2014). When lignin is degraded during the papermaking process, a lot of phenols are released into the environment (Ansah, 2015). On top of that, nitrophenols are created photochemically from airborne car emissions. Ansah (2015) explains that phenols can also be generated via the breakdown of organophosphorus (Glezer, 2003), insecticides, and chlorophenoxyacetic acids.

Since it can be used for a variety of purposes but also poses risks to human health and the environment, it is considered a hazardous substance. The pulp and paper industry's wastewater contains phenol since the chemical is extremely soluble in water (Khieu et al., 2009). The discovered catalyst will aid in the conversion of phenol to diphenols, which have a wide range of uses including skin therapy (Ishack and Lipner, 2022) and as photographic chemicals, polymerisation inhibitors, antioxidants, and flavoring compounds (Adam et al., 2010).

1.3 Objectives of the study.

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

1) To extract the silica from rice husk by using the sol-gel method.
2) 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).
3) To study the catalytic activity of silica on the oxidation of phenol by using various mass of catalyst.

1.4 Scope of the Study

This study aims to learn more about the processes involved in phenol oxidation and silica extraction from RH. The catalyst is safe for the environment because to the silica it produces from RH. To begin this inquiry, silica will be extracted from RH, and the weight difference will be determined. Next, the catalyst's performance on phenol oxidation will be assessed by varying operating conditions. The phenol oxidation reaction's conclusion will determine the catalyst activity and conditions required for optimal reaction performance.

1.5 Significant of Study

This research employed RH-collected silica to create an organic catalyst effective at oxidizing phenol to its products, thereby mitigating the chemical's potential to harm aquatic ecosystems and human health. The generated catalyst is safe for the environment and helps boost reaction speed and product yield. The proposed method allows for RH to be used in its entirety with zero negative effects on the surrounding ecosystem. To maintain thriving ecosystems, this study is essential for humanity and the planet.

Using rice husk as a source of silica can also have significant environmental benefits. Rice husk is a waste material produced in large quantities in rice-producing countries, and its improper disposal can lead to environmental pollution and health hazards. Utilizing this waste material for extracting silica, it reduces the volume of waste, promotes sustainability, and helps reduce the environmental impact of rice farming.

Finally, the study of extracting silica from rice husks can also contribute to the development of new technologies that can efficiently extract silica from other types of agricultural waste. This can lead to the production of materials that are eco-friendlier and more sustainable, thus helping to reduce the negative impact of human activities on the environment.

Overall, studying the extraction of silica from rice husk has many practical applications, and environmental benefits, and can contribute to the advancement of sustainable technologies.

CHAPTER 2

LITERATURE REVIEW

2.1 Rice Husk

RH is a component of the vegetation Oryza sativa's body. It is also known as rice hull, which is the natural casing of rice kernel or seed that protects it during the growing season. RH is a byproduct of agriculture prevalent in rice-producing nations. It results from the cultivation and production of rice. Asia produces and consumes approximately 90 percent of the world's rice, making it the most significant staple food in the region. On 29 million hectares, China remains the world's largest producer, producing one-third of Asia's total. On 43 million hectares, India produces approximately one-fourth of the world's food. In Asia, the countries that produce the most rice are Indonesia, Bangladesh, Vietnam, Thailand, Myanmar, the Philippines, and Japan. Average yields in these countries range from 2.6 t ha-1 to 6.5 t ha-1 (Barbier, 2017). Rice husk comprises cellulose (50%) and lignin (25-30%), silica (15-20%), and water (10-15%) and accounts for approximately 20% of the weight of rice. The bulk density of rice husk ranges between 90 and 150 kg/m3.

Rice husk ash (RHA) is found in rice-growing regions such as China, India, and the Far East. RHA is a byproduct of burning rice hulls. After burning the preponderance of rice husk's evaporable components, silicates are the most abundant byproduct. The properties of the ash are determined by (1) the rice fibre content, (2) the burning temperature, and (3) the burning time. Every 100 kg of husks burnt in a boiler for example will yield about 25 kg of RHA (Alengaram, 2021). Malaysia is also one of the rice-producing nations. Rice is the most important commodity in Malaysia because it has long been their staple food. The aggregate production of paddy and rice in Malaysia is increasing over time. According to data from the Malaysian Department of Agriculture, rice production will increase by 2.5% between 2018 and 2020, benefiting Malaysia's agricultural sector. As rice production increases, so does the proportion of RH produce. RH is utilised predominantly as a fertiliser, planting medium to improve soil quality, insulating material, and fuel, and is primarily burned to produce ash. Biodegradation of agricultural waste into compost and incorporation into soil may enhance the nutrient recycling and maintain soil fertility (Thiyageshwari et al., 2018).

Several studies of RH have been utilised in various research fields, including water treatment, as an alternative material in a cementitious matrix by producing sustainable cement-based material, as fuel in producing stream in parboiling process, as source of silica in ceramic production, upgrading soil properties for agriculture by removing heavy metals, reducing soil bulk density, and increasing nutrients, among others.

Utilising RH eliminates the waste disposal issue associated with RH and generates a valuable commodity. Currently, it is an essential catalyst in the chemical industry and a basic material in the production of silicon. Comparatively, silica recovered from RH is significantly superior and more cost-effective than silica produced from quartz using current technologies. The method for extracting silica from RH uses considerably less energy and is more energy-efficient than the method that involves fusing sand of a particular quality.

2.2 Silica

The chemical formula for silicon dioxide SiO_2 or silica consists primarily of oxygen atoms with trace quantities of hydrogen and silicon atoms. It is one of the two most prevalent substances on Earth. Silicon dioxide (SiO_2) occurs naturally in the Earth's crust and atmosphere in the form of quartz, which is typically crystalline and infrequently amorphous. The vast majority of the minerals that make up the rocks of Earth's crust are silicate minerals (Earle et al., 2015) . Over 90% of the main component of minerals and rocks on Earth is silicate, which is primarily ionic. Natural sources of silica include sand, vegetation, sponges, and animals. Additionally, hot springs in particular regions generate a silica solution that is supersaturated. Grass, grains, and rice absorb silica and deposit it in their tissues as minute amorphous opaline particles, which are subsequently detected in the soil and the intestinal tracts of grazing animals. Silica from beach sand or quartz can only be extracted in limited quantities, as it dissolves very slowly and in minute quantities. Silica is perpetually extracted from saltwater by biochemical processes, and silica accumulated within diatoms, sponges, and plants is expelled. The increased pressure in deep seawater may make amorphous silica more soluble in it. The solubility of amorphous silica in seawater at 0°C and from 1 to 1,220 atm (Willey et al., 1974)

Temperature is the most significant factor in obtaining amorphous silica from relative humidity because silica crystallisation can be controlled by both temperature and burning duration. The carbon residues cannot be eliminated by reheating the ash, which would require more time and higher temperatures, and the silica will transform into a crystalline form. Amorphous silica is silicon dioxide with no crystalline structure and a random arrangement. In Figure 2.1, the structural configurations of amorphous and crystalline silica are compared. In the crystal structure of quartz, the silicon atom is bonded to four oxygen atoms tetrahedrally.

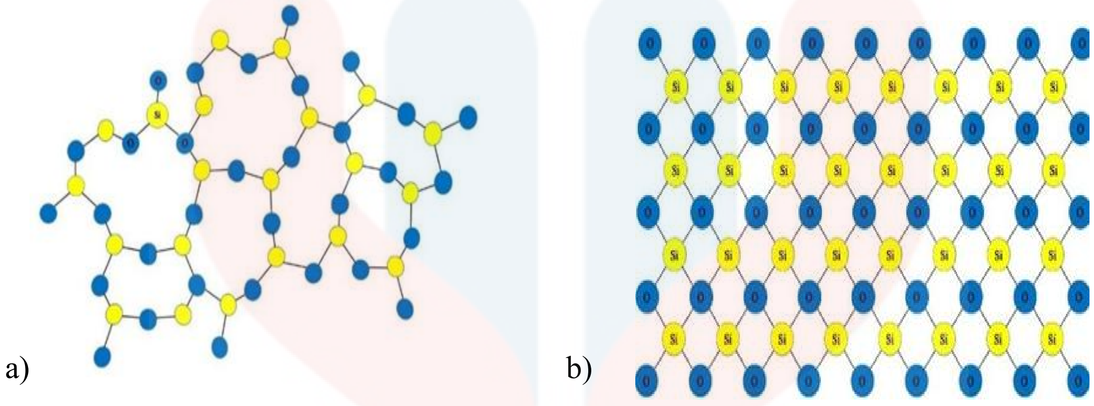

Figure 2.1: a) amorphous silica structure and b) crystalline structure

Table 2.1: Comparison between amorphous silica and crystalline silica

Description	Amorphous silica	Crystalline silica
Structure arrangement	Amorphous silica refers to silicon dioxide that lacks long-range order in its atomic structure.	Crystalline silica is characterized by a highly ordered atomic arrangement in a repeating three-dimensional lattice structure.
Porosity	High porosity 50-100 nm	Low porosity
Solubility	Amorphous silica exhibits higher solubility in water due to its non-crystalline structure	Crystalline silica, particularly in its most common polymorph quartz, is considered relatively insoluble

		in water under normal conditions.
Used in	as fillers in the rubberindustry, as free-flow and anti-caking agents in powder materials, as liquid carriers, uses in toothpaste additives, paints, silicon rubber, insulation material, adhesives, printing inks, plastisol car undercoats, cosmetics and many others.	It is also used to make products such as glass,pottery, ceramics, bricks,and artificial stone
Toxicity	Some of the benefits of silica include low toxicity ofthe amorphous oxide.	

According to previous research, amorphous silica has a porous structure. As a catalyst for the development of green chemistry, it is well-suited for functionalisation with organic compounds (Kadja et al., 2022). The properties of silica are essential to its applications, each of which requires a distinct set of properties. Regarding the catalyst, it must possess an amorphous silica structure, a large surface area, and other characteristics that promote high catalytic activity. Numerous forms of silica, including mcm 41, mcm 48, sba 15, sba 16, HMS, and others, have been synthesised and are currently being investigated by scientists. Each possesses both benefits and drawbacks.

2.3 Phenol

Phenol (C_6H_5OH) is 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 is 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 is phenol (Smith et al., 1969). Since phenol is 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).

The diverse range of applications involving phenol may result in the release of elevated phenol levels into the environment, posing a significant risk to ecosystem quality. This can disrupt nutrient cycling and negatively impact biological ecosystems, as phenol is recognized as one of the most hazardous chemicals.

CHAPTER 3

METHODOLOGY

MATERIAL 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 step 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 step is the treatment in RH. A sample of 30.0g (RH) was weighed, and 24 hours were spent stirring it with 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 step is the preparation of sodium silicate solution. The preparation of sodium silicate solution, which are 3.0 g of RHA mix was combined with 500 ml 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.

3.2.3 The characteristic of the catalyst

Following its manufacturing, the catalyst underwent 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).

To begin, thermogravimetric analysis (TGA) was employed to determine the catalyst's properties. TGA was a controlled thermal analysis method for studying how a sample's weight changed over time or in response to variations in temperature. By monitoring the rate of change in mass as a function of temperature, TGA may ascertain if a material is thermally stable. By studying patterns of weight loss, TGA is able to determine the chemical make-up of complicated materials. Decomposition or volatilization of a mixture's components at different temperatures makes it possible to identify and quantify those components. To produce a different result in the same analyzer, TGA analysis was done in combination with Differential Scanning Calorimetry (DSC). Chemical and physical changes to materials may be studied by measuring the heat flow as a function of temperature using differential scanning calorimetry (DSC), a thermal analysis technique. Crystallisation, melting, glass transitions, and polymorphic transitions are all examples of phase transitions that were extensively studied using DSC. Decomposition spectroscopy (DSC) may also reveal material thermal stability by revealing deterioration or decomposition events as temperatures rise.

Second, Fourier Transform Infrared Spectroscopy was the method utilised to determine the catalyst's properties. In the past, Fourier transform infrared spectroscopy (FTIR) was a popular analytical tool for determining a material's molecular make-up, bonding relationships, and chemical composition. In this test, the chemical interactions between the components were investigated using Fourier transform infrared spectroscopy (FTIR) on infrared light. Finding the chemical functional groups present in the sample's RHA was the goal of the FTIR analysis. In this investigation, the scanning range was 4000-500 cm-1.

Furthermore, Power X-ray diffraction (XRD) was the method employed to determine the catalyst's properties. X-ray powder diffraction (XRD) was a method for analysing crystalline materials at the atomic and molecular level. The properties of the unit cell, crystallite size, crystallinity level, and phase purity were all determined using XRD. The XRD process included precisely angling a beam of light at the sample. The inter-atomic lengths in a sample determine how the X-rays deflect or diffract.

Lastly, scanning electron microscopy (SEM) was employed to examine the catalyst's properties. Surface morphology, topography, and material composition might be seen at high magnifications and resolutions using SEM, a potent imaging technology. Under greater magnification optical microscopy, crazing patterns on the surface of the component were examined using SEM. Scanning electron microscopy (SEM) revealed the surface porous catalyst structure of the sample and verified that it was in an amorphous state devoid of crystal structure. The porous nature of the material was shown during both low and high magnification scanning electron microscopy examinations of a finely ground sample. An analytical method called energy dispersive X-ray analysis (EDX) was utilised alongside scanning electron microscopy (SEM) to offer elemental analysis of materials. The elemental composition of the sample was assessed using EDX. Spreading the sample powder as thinly as possible, it was attached to a stub of SEM using conductive adhesive tape. The crazing patterns of the sample and the average value of the elemental analysis could be found.

3.2.3 The catalytic activity reaction in oxidation of phenol

The activity of the catalyst will be assessed in the oxidation of phenol. A 250 ml round-bottom flask with two necks and a condenser that cools water. In a standard run, 20 mmol of phenol was dissolved in 20 ml of acetone. Before being cooled in a desiccator to decrease moisture content, the catalyst was heated to 100°C for 24 hours to activate it and remove any water molecules that had been adsorbed. The 0.1 g of active catalyst and the mixture were placed in a round-bottomed flask and heated to 75°C in an oil bath. The reaction mixture was agitated at 600 rpm and 20 mmol of hydrogen peroxide (H_20_2) was added dropwise after the reaction temperature had stabilised. Four hours passed during the answer. Half a millilitre was removed and filtered using a syringe at the hourly intervals. The sample was internally standardised with 20µL of acetophenone before GC-MS analysis. The solvent, temperature, time, catalyst mass, and phenol-to-catalyst molar ratio were some of the variables employed to regulate the catalytic process.

A potent analytical tool, gas chromatography mass spectrometry (GC-MS) could separate, identify, and measure chemicals in complicated mixtures. The different goods' identities were confirmed by GCMS. It gives comprehensive data on sample structure and composition by integrating gas chromatography (GC) and mass spectrometry (MS). The items were double-checked by comparing the GC of the individual pure hydroquinone. The compounds were subjected to chemical ionisation (CI) or electron impact (EI) procedures in the MS, which led to the creation of charged fragments.

Phenol oxidation affects the response to various solvents. We used a range of solvents, including methanol, toluene, and acetone, to find out which one gives the greatest catalytic results. Constant ratios of 20:20 mmol, a mass constant of 0.10 g, and a temperature of 75°C for three hours were all part of process 3.5.1. Filtering the reaction mixture to remove the catalyst is an important step once the reaction is complete. The data display made use of average values, and every reaction was performed three times.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Characteristics of catalyst

All the catalyst's attributes were established to yield results from any testing device used to study the catalyst in the sample. To investigate these properties, four techniques were used: thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), Fourier Transform Infrared Spectroscopy (FTIR), power x-ray diffraction (XRD), scanning electron microscopy (SEM), and energy dispersive x-ray (EDX).

4.1.1 XRD result

With the help of the XRD equipment, the x-ray diffraction pattern of the RHA-created silica was shown. Researchers can determine the types and amounts of crystalline phases in a silica sample by comparing the recorded diffraction peaks to reference patterns for known crystal structures. Different crystal phases can lead to different material properties, and XRD provided a non-destructive way to examine the crystal structures of composite materials (Polini & Yang, 2017) Lattice parameters, thermal expansion coefficient, residual stress and strain, crystalline structure, size, and orientation, dislocation density, phase identification, quantification, and transformation, and XRD patterns are all useful tools for materials science. One example of how data may be used to tailor materials to specific applications is by increasing the mechanical strength of composites (Ali et al., 2022).

Following the separation of the silica powder from the resulting ash, it underwent a titration using a solution of hydrochloric acid (HCl) followed by treatment with a sodium hydroxide (NaOH) solution. The results obtained from the XRD analysis indicated that the extracted silica powder exhibited an amorphous structure, signifying that it maintained the maximum quantity of its active properties. As stated in the study by Chik et al. (n.d.), the rationale behind the selection of amorphous silica for the production of silicon-based compounds is as follows. Within the scope of this discourse, the expression "active" pertains to the amorphous state of silica. This state is more chemically reactive or readily engages in chemical reactions when compared to its crystalline counterpart (Nzereogu et al., 2023). The characteristic reactivity of amorphous silica is commonly attributed to its disorderly and reactive surface structure. The knowledge of crystalline phases in a silica sample provides valuable insights for optimizing production processes, tailoring material properties, and ensuring the suitability of silica for specific applications across various industries. It contributes to both the advancement of materials science and the practical utilization of silica in diverse fields (Kniess et al., n.d.).

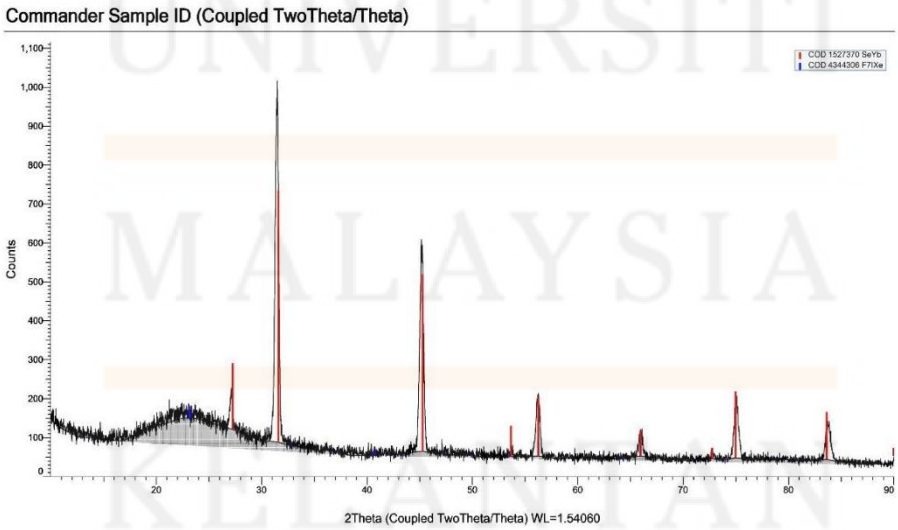

Figure 4.1: The X-ray diffraction pattern for RHA

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

SAMPLE	AMORPHOUS (%)	CRYSTALLINE (%)
RHA	43.4	56.6

Figure 4.1 shows the process of using hydrochloric acid-made X-ray diffraction patterns to confirm the genuineness of the SiO_2 samples. There were absolutely no indications of building on the huge, flat peak of the hill. The amorphous peak can be observed at 2θ-31° in the XRD pattern. The XRD pattern showed that silica is amorphous. Table 1 reveals that crystalline silica made up around 56.6% of the total, whereas amorphous silica accounted for about 43.4%. According to (Gutiérrez-Castorena & Effland, 2010), amorphous silica originated from SiO_2 tetrahedra that are organised in two or three layers on cristobalite and tridymite, which are neighbours to crystalline SiO_2 polymorphs. Without the crystal structure that allows for long-range interactions, amorphous solids show entanglements or chemical bonding (Schmitz, 2017). The XRD pattern of hydrochloric acid-treated silica RHA shows a broad hump for amorphous materials and sharp diffraction peaks for crystalline ones.

Silica extraction from RHA required the removal of amorphous silica due to its higher degree of activity compared to crystalline silica (Nayak et al., 2019). Crystalline silica had better mechanical properties and performed better in non-distractive tests than amorphous silica.

4.1.2 Fourier transformed infrared spectroscopy analysis (FTIR)

FTIR spectroscopy was used to analyse chemical changes on the surface of steam explosion-pretreated rice husk (Latika B and Dilip Kumar S). As can be shown in Figure 4.2, the measured bandwidth between 3750.27cm-1 (RHA) belongs to the isolated and surface functional O-H groups of (Si-OH) in the silica as well as the water molecules that have been adsorbed on the silica surface.

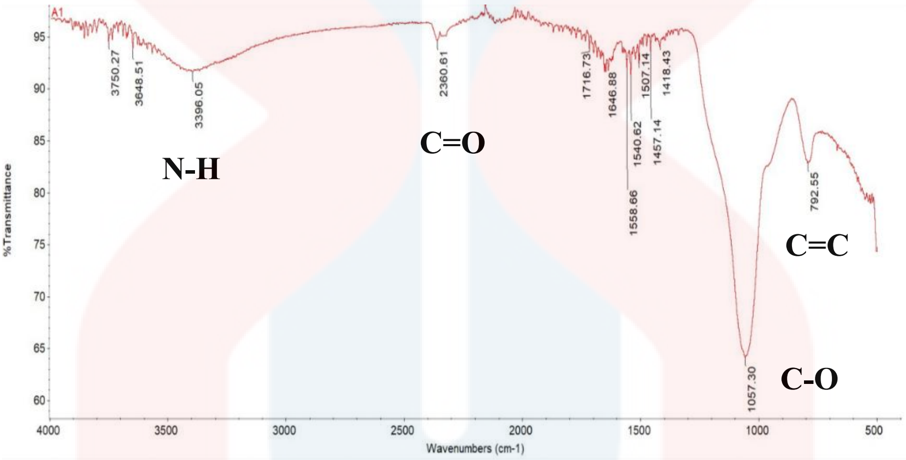

Figure 4.2: The FT-IR spectra of RHA

The bandwidth recorded between 3750.27cm^{-1} RHA, as shown in Figure 4.2, was caused by both surface functional O-H groups (Si-OH) in the silica and adsorbed water molecules on the silica surface. According to the N-H group, an absorption band centered at 3396.05 cm^{-1} indicates the presence of adsorbed water molecules between 3500 and 3000 cm. A C-O wavenumber of 2360.61 cm^{-1} is characteristic of rare earth halides (RHAs). In the range of 2500 to 2000 cm^{-1}, the performance was at its highest at 2113.01 cm^{-1}. Peaks were seen at 1540.62 cm^{-1} before 1500 cm^{-1}. In the asymmetric stretching vibrations of the Si-OH band, the vibration of the oxygen atom is coupled with the vibrations of the surrounding atoms (Pallavi et al., 2012). The Si-O-Si bond's bending, symmetric, and asymmetric stretching vibrations are caused by the bands in Figure 4.5 spectra, which are 1057.30 cm^{-1} for C-O and 792.55 cm^{-1} for C=C. According to Samah et al. (2020), the base treatment removed inorganic components from the surface of the rice husk, which resulted in a less intense silica functional group (Si-O-Si) in the chemically treated sample compared to the raw husk.

4.1.3 Thermogravimetric Analysis (TGA) with Differential Scanning Calorimetry (DSC)

The RHA powder sample underwent TGA, and its weight was determined using the TGA/DSC 2 HT Mettler Toledo. The sample weighed 0.0026 grams. In the pin-holed aluminum crucibles, the TGA process was adjusted to heat at a rate of 10°C/min from 40°C to 600°C while purging nitrogen gas at a rate of 20 mL/min. The RHA TGA thermogram revealed a decrease in the weight loss of the sample. When the fexofenadine hydrochloride (FEX) began to melt at 43.74°C, resulting in an 8.8029% mass loss, the weight loss process started. There was no dehydration before the temperature reached the melting point. A highly reactive RHA is produced by burning rice husk at regulated temperatures.

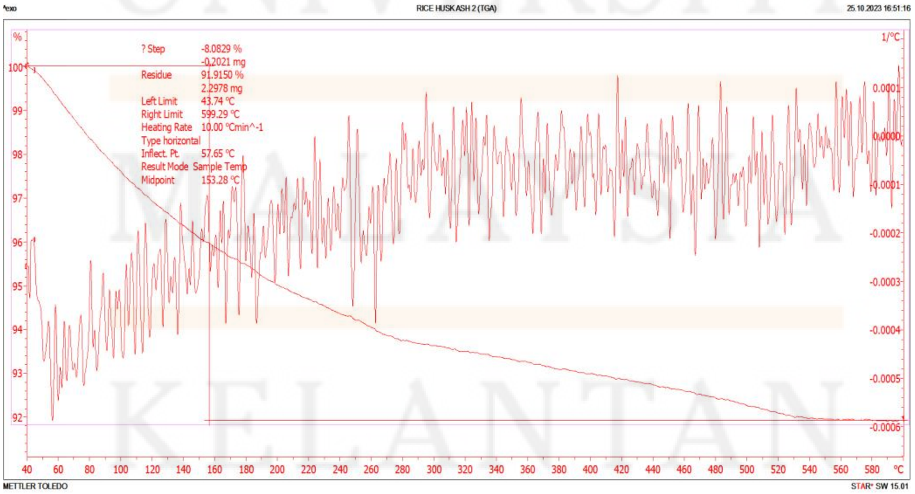

Figure 4.3: TGA thermogram of RHA

The reactivity of the interacting materials is enhanced as their fineness increases (J et al., 2012). The TGA test result displayed a drying or desorption on the graph of TGA curves in Figure 4.3. This curve develops during particular procedure where the mass weight of RHA was connected to non-volatile components or breakdown processes that occur at higher temperatures. The TGA curves in single stage decomposition and was based on inflection points, mass loss rates, and residual masses, according to (Halim et al., 2018) aids in a more complex interpretation of the findings. Weight decreases were seen in RHCI, as shown by the TGA thermogram. The melting point of RHA is 43.74 degrees Celsius, which results in a mass loss of 8.0829%. There were no indications of dehydration to be seen prior to the temperature beginning to melt. There is a downward trend seen in Figure 4.3. There is a possibility that this kind of curve will be produced when the increase in weight of the sample is brought about by surface oxidation processes that take place in an environment that is reacting.

For another research, There were some factor that affecting the TGA result. The first factor affecting TGA result was a weight of sample.

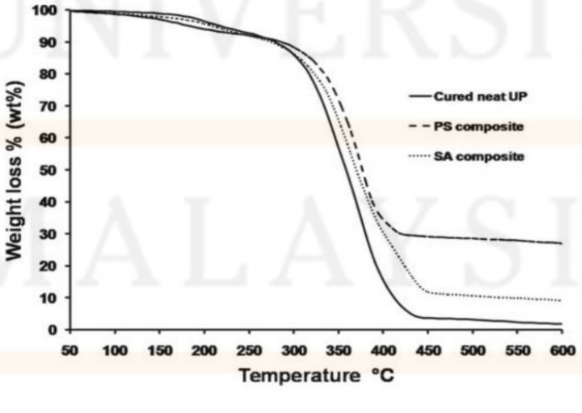

Figure 4.4: Thermogravimetric TGA curves by RHA on precipitated silica (PS) and silica aerogel (SA) (Zulhelmi et al, 2018)

The TGA result in the weight of sample has showed different between Figure 4.3 and Figure 4.4 which was 2.6 mg and 10 mg. This was mainly because the initial amount in the sample seen in Figure 4.3 is so little and easily burned. Figure 4.4 shows a different sample that differs in that it contains silica aerogel (SA), which has a higher amount and may be burnt for a longer duration in TGA during melting. The TGA result was determined by the second portion of the equation, which included the temperature and the rate of heating. Figure 4.3's sample has lower temperatures and a higher heating rate, leading to faster desorption and possibly affecting the form and characteristics of TGA curves. This is why this happened. In contrast, Figure 4.4 demonstrates that the heating rate and temperature are most affected by the weight sample on precipitated silica (PS) and silica aerogel (SA). The finding in Figure 4.3 should be interpreted as a decrease in RHA crystallinity proportional to weight loss when the maximum temperature is set at 1200 degrees Celsius, according to Ramadhansyah et al. (2012). Moreover, the diverse materials used to construct the sample also impacted the TGA outcome. The rationale behind this, as stated by Zulhelmi et al. (2018), is that the RHA sample in Figure 4.3 did not include any additional material or chemicals. In contrast, the TGA results for the sample depicted in Figure 4.4 showed that the RHA had been mixed with unsaturated polyester (UP) resin on amorphous precipitated silica (PS) and silica aerogel (SA).

DSC curves of RHA sample in Figure 4.3 showed a heat flow rate in 20mL/min and the temperatures range from 40°C to 600°C.

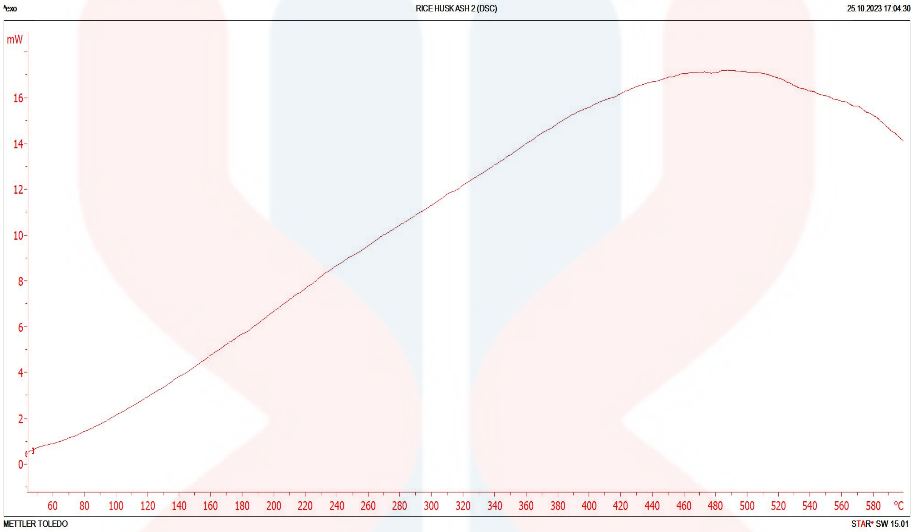

Figure 4.5: DSC analysis treated by RHA

Due to the DSC curves, exothermic curves played a major role in the experiment. As soon as the heat flow rate rose over the baseline, the exothermic process began when the sample developed heat. This marked the beginning of the procedure. A more complex chemical route may have been indicated by the greater exothermic peak shape, as shown in the study by (Daud et al 2020). A faint and broad peak at lower temperatures and a sharp and powerful peak at higher temperatures are the usual peaks seen in differential scanning calorimetry (DSC) (Adam West, 2018). At lower temperatures, you can usually see the smaller peak. The exothermic peak, which started at 40 degrees Celsius and persisted until it reached 520 degrees Celsius, was the final peak that showed some process-related crystallisation temperatures. The data may be acquired by calculating the glass transition temperature (Tg) and the stiffness of composites, according to a study by (Syifa et al 2020). The nucleation process occurred during the early stages of the material's growth at the RHA, as reported by (Russo et al. 2013), who looked at the reaction on crystallisation. The change from an amorphous to a crystalline state is often accompanied by an exothermic reaction. After the treatment was finished, the endothermic study showed that the heating rate slowed down to temperatures between 520 and 600 degrees Celsius.

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

The use of scanning electron microscopy (SEM) allowed for the microscopy of RHA functionalized with RHA to be examined. You may see RHA's scanning electron micrograph in Figure 4.6. At low magnification, this micrograph shows that RHA's topology consists of agglomeration and a few surface particles with a spherical shape. Uneven morphologies and tiny particle sizes are seen on the RHA surface. These features are plain to see. Because of its limited surface area and lack of porosity, RHA was found to contain particles with smooth surfaces. The RHA surface reveals that it is made up of small particle sizes randomly organized with irregular morphologies. Uneven rock-like particles. Porosity and low surface area of RHA is caused by certain particles with smooth surfaces and surfaces porous.

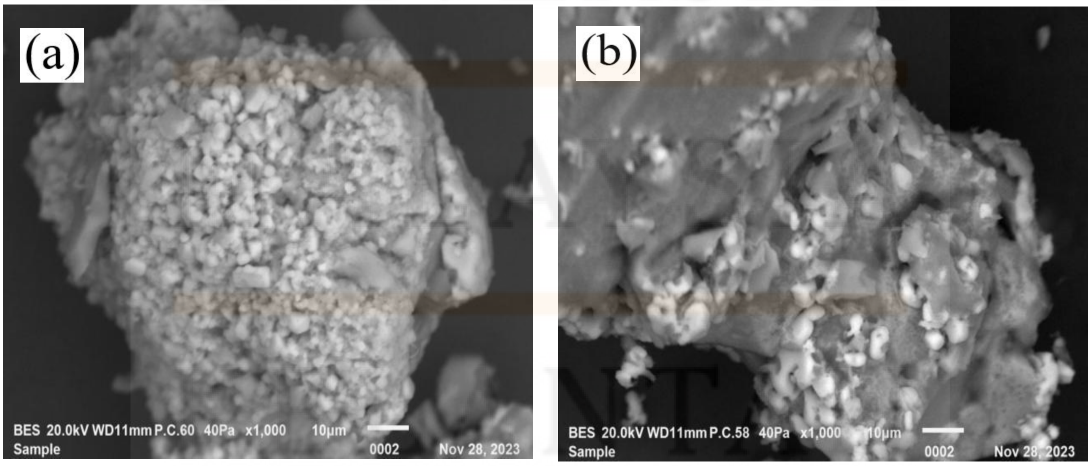

Figure 4.6: The SEM image of RHA (a) and (b)

Another study is "Preparation and characterization of high-purity amorphous silica from rice husk" (Ngoc et al., 2018). See surface morphology (SEM pictures) in Figure 4.6A for silica samples prepared by alkaline extraction and in Figure 4.6B for silica samples prepared by SiO2-precipitation.

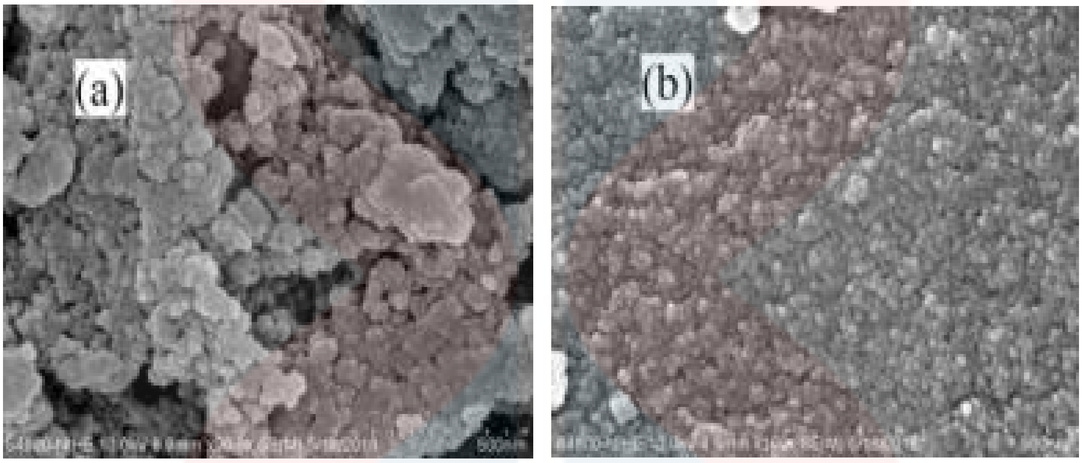

Figure 4.7: SEM images of silica produced from alkaline-extraction (a) and SiO_2-precipitation methods (b)

The silica products are in the form of aggregates of primary particles. The alkaline-extracted silica has voids and pores (or porous structure) despite its bigger primary particles (about 50 nm) compared to SiO2-precipitated silica (about 25 nm). This observation from SEM images agrees well with the measured BET surface areas of 186.5792 and 68.2269 (m^2/g) for alkaline-extracted and SiO2-precipitated silica, respectively.

On the other research, an alternative method of investigation involves the creation of amorphous silica using alkaline extraction, using reflux and subsequent acidification with either acetic or hydrochloric acid (Dhaneswara et al., 2020) . The surface morphology of amorphous silica was examined by analysing scanning electron microscope pictures. The images were taken at magnifications of 50,000× and 200,000×.

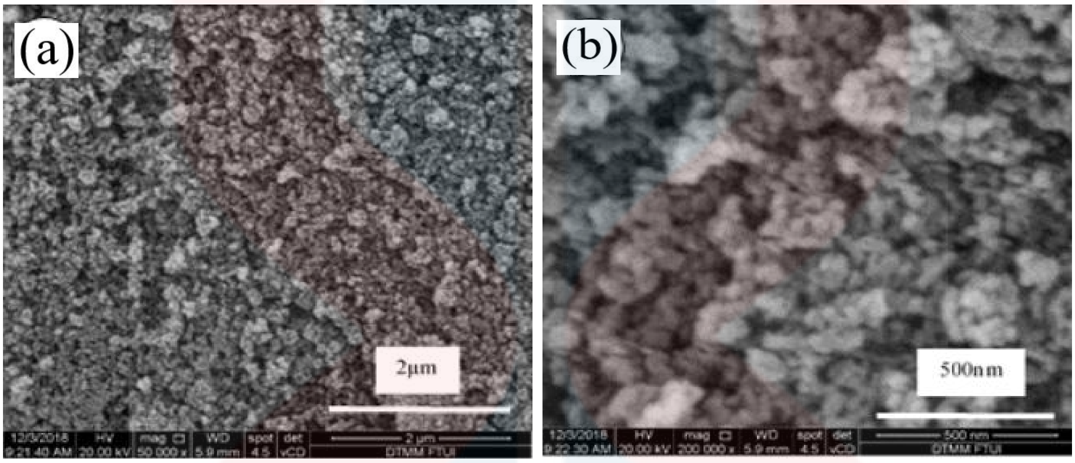

Figure 4.8: SEM micrograph of silica from rice husk using magnification:(a) 50.000× and (b) 200.000×

Figure 4.8A shows that the grain of the synthesized silica from rice husk has an irregular shape and a size of 9–12 nm. Figure 4.8B, which presents a higher SEM magnification, clearly shows that grain sizes and of varying sizes can be clearly seen, which indicates that the synthesized silica can be identified as heterogeneous mesopore silica. The brightly colored surfaces indicate amorphous silica, whereas the dark-colored surfaces indicate pore cavities. This is supported by the data in Figure 3, which shows that the synthesized silica is a type IV silica with a mesoporous structure. However, the structure of the synthesized silica shows a tendency to agglomerate.

RHA elemental studies were performed using the EDS at the specified point in the SEM micrograph shown in Figure 4.9 and Figure 5.0.

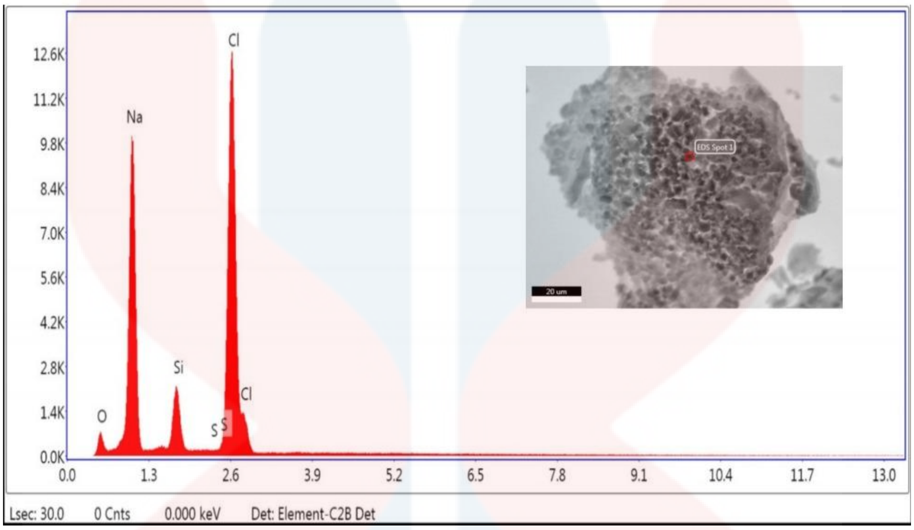

Figure 4.9: EDX profile of RHA with SEM micrograph of RHA at spot 1

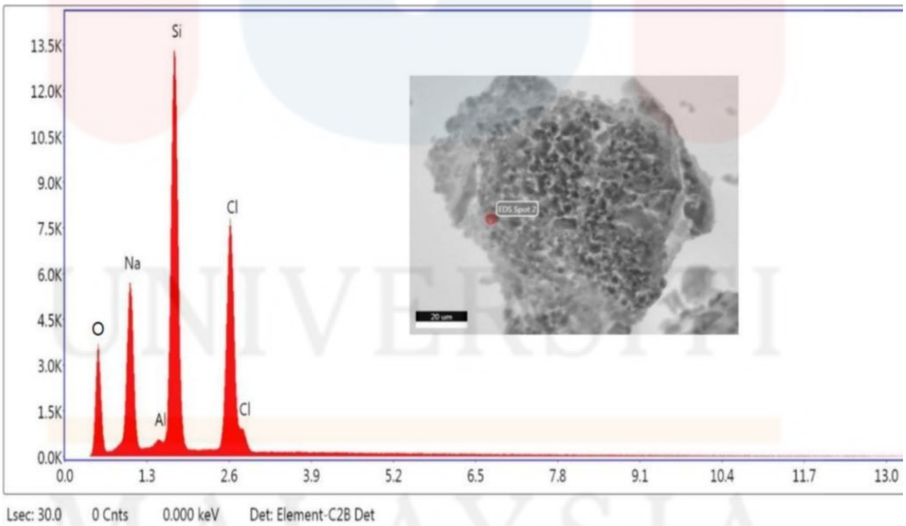

Figure 5.0: EDX profile of RHA with SEM micrograph of RHA at spot 2

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

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

The elemental analyses of RHA were carried out by the EDS at the particular region in the SEM micrograph that is depicted in Table 4.2. The RHA at position 2 reveals that Si and Cl have a higher weight percentage than Na and O, which are only 22.22% and 15.65% respectively. Si and Cl have a weight percentage of 40.47% and 39.54% respectively. The weight percentage of sodium is 38.61%, the weight percentage of chlorine is 37.90%, and the weight percentage of oxygen is 13.61%. In contrast, the weight percentage of silicon is only 9.74%, and the weight percentage of sulfur is 0.14%.

4.1.5 Catalytic activity of RHA in Oxidation of Phenol

Phenol was oxidised with hydrogen peroxide as the oxidising agent, and a variety of criteria were used to assess the RHA. This was done so that the catalytic activity of the catalysts that were created could be evaluated. The investigation of the components of the reaction in connection to the effects that different solvents have on the reaction. During the process of oxidation, the primary molecule that was produced was phenol, 4-phenoxy, also known as hydroquinone monophenyl ether and acetic acid, phenyl easter, and phenyl-β-d-glucopyranoside. In addition, there were instances where a small by-product was produced.

The reactions were examined with the catalyst masses set at 0.10 grammes, 0.02 grammes, and 0.03 grammes, while the remaining parameters were maintained at the same levels (temperatures ranging from around 65 to 75 degrees Celsius, a molar ratio of phenol to H_2O_2 of 1:1, and a reaction time of three hours).

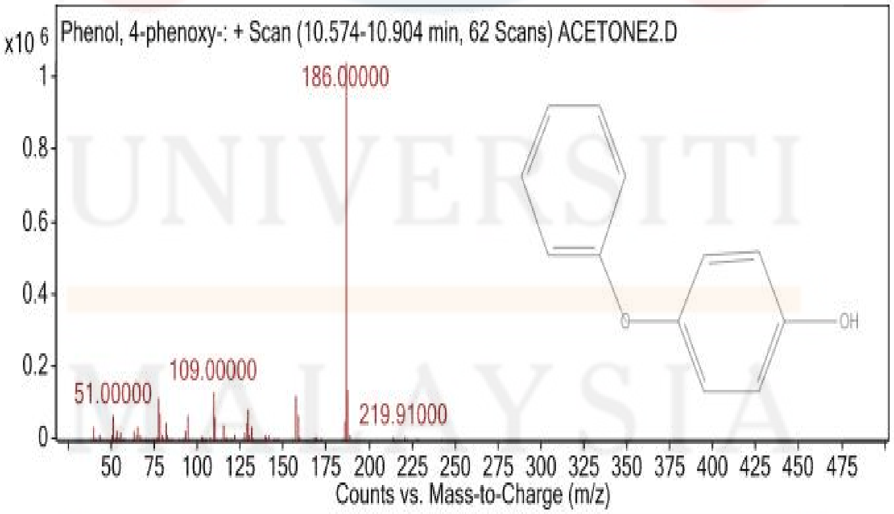

Figure 5.1: Shows the major molecule formed.

As shown in Figure 5.1, the most important molecule that was produced was phenol, 4-phenoxy-, which is also referred to as hydroquinone monophenyl ether. According to Sarkar et al. (2013), hydroquinone (HQ) was a dihydric phenol that possessed two important derivatives. These derivatives were considered to be monobenzyl and monomethyl ether of hydroquinone. In addition to its application as a stabiliser in paints, varnishes, lubricants, and motor fuels, it may also be utilised as a dying intermediate and a developing agent for photographs. In addition to that, it is utilised in the production of methacrylic, various acrylates, vinyl acetate monomer (VAM), styrene monomer, and unsaturated polyesters. Hydroquinone was utilised in commercially available cosmetic skin lightening formulations in the countries that make up the European Union from the beginning of the 1950s until the year 2001. Beginning in the 1960s, it was also offered as a medical medicine. Additionally, it is used into cosmetic formulations utilised for the purpose of coating fingernails and colouring hair (Francisco and Ana, 2013).

A minor by-product that showed up is phenyl ester.

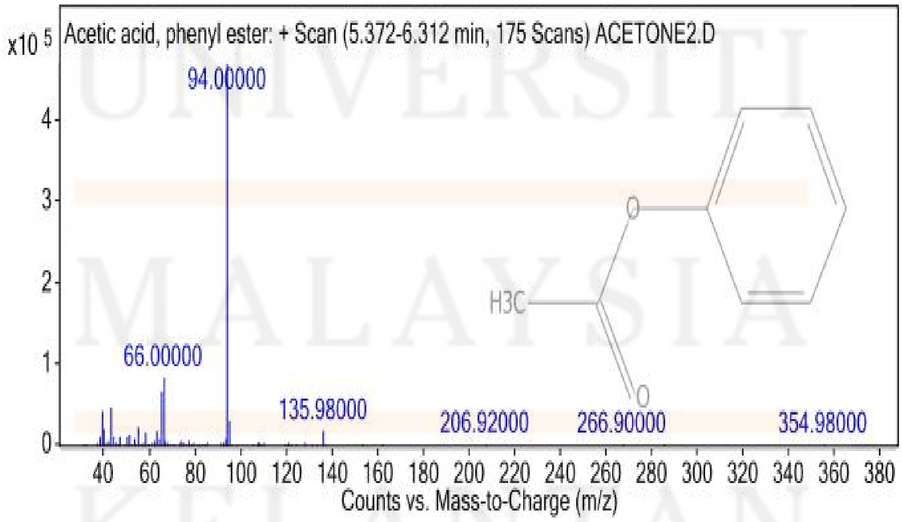

Figure 5.2: Shows the minor by-product formed.

The substance that was seen in Figure 4.15 was phenyl ester of acetic acid, which is also referred to as phenyl acetate. A phenyl ester is a kind of chemical molecule that is identified by the presence of a phenyl group, which is a benzene ring, that is connected to an ester functional group. Using a treatment with one equivalent of hydrogen peroxide at a pH of 10.5 in a range of solvents, such as 80% acetone, dimethylfonnamide, hexamethylphosphoramide, or trifluoroethanol, it is possible to remove it selectively. Phenyl esters are employed extensively in the fragrance and flavour industries for the purpose of imparting different scents or tastes of their products. According to Arceli et al. (2003), it has the ability to improve the aromatic and flavorful aspects of food products, particularly fragrances and colognes.

One type of phenolic molecule that may be discovered in the cell walls of plants is called phenyl-beta-D-glucopyranoside.

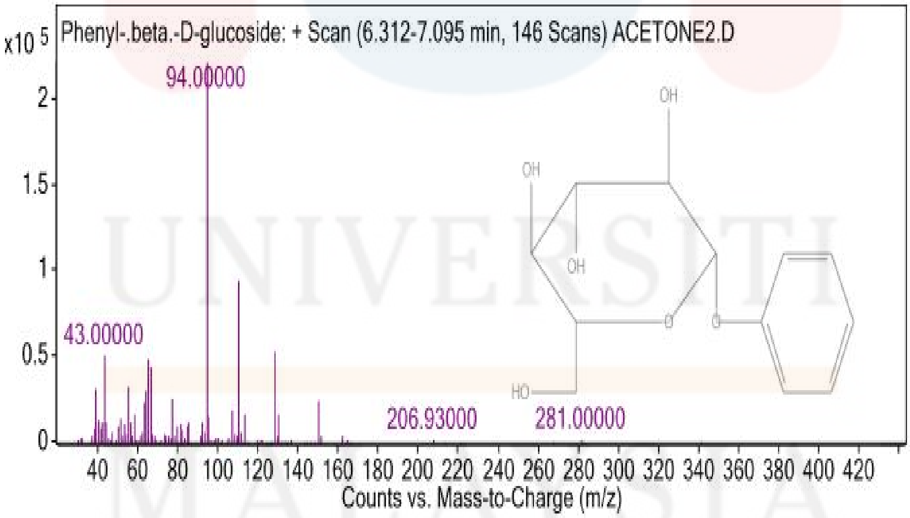

Figure 5.3: Shows the minor by-product formed.

It is a monosaccharide that has an ester bond, and esterases are responsible for hydrolyzing it in order to produce protocatechuic acid. In addition to being an inhibitor of sugar transport, phenyl-beta-D-glucopyranoside has been demonstrated to have protective effects on the liver in some circumstances, such as when it is exposed to hazardous compounds. When this sugar attaches to proteins, it has the potential to change the function of the proteins that it binds to ((Jockusch et al., 2003). Phenyl-beta-D-glucopyranoside has been demonstrated to block glycolysis in hepatic tissues, and tannins are produced from erythrocytes that have been treated with this chemical. Despite being exposed to acidic environments, phenyl-beta-D-glucopyranoside has been demonstrated to maintain its chemical stability.

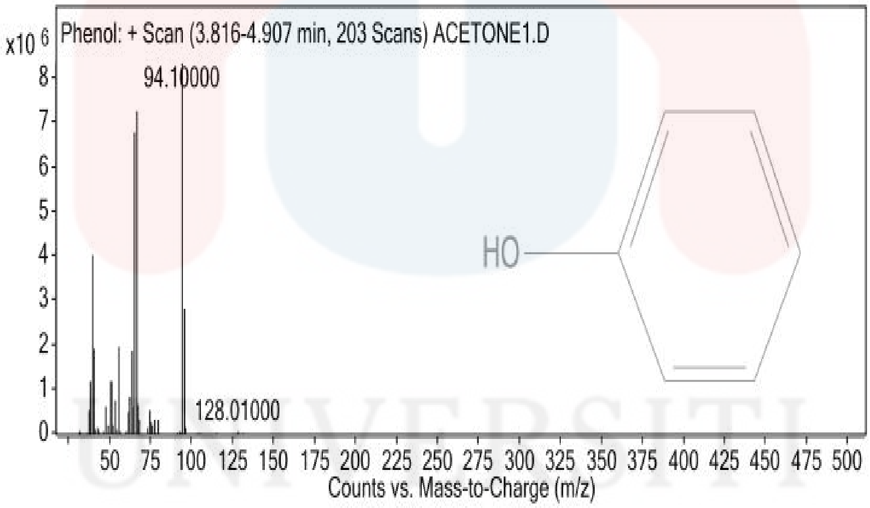

Figure 5.4: Shows mass-to-charge ratio (m/z) at 0.10g reaction.

The most prominent peak, which is located at a mass-to-charge ratio of 94.1000, shows that the ion that is the most numerous among those that have been observed. The other one is at a mass-to-charge ratio of 128.01000, which indicates that it is composed of ions with those particular ratios. Molecular ion of phenol

(C6H5OH) with a molecular weight of 94 might be represented by the signal at m/z 94.1000, which is a possible interpretation.

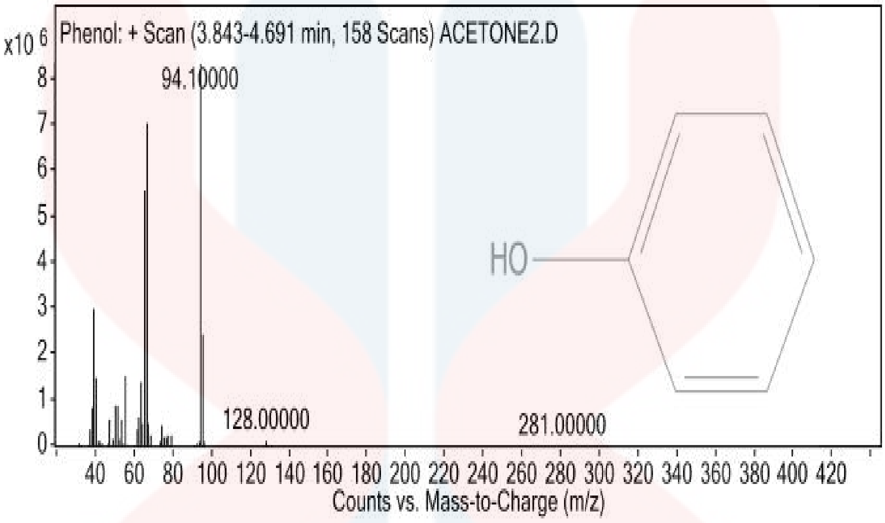

Figure 5.5: Shows mass-to-charge ratio (m/z) at 0.20 reaction.

It is the molecular ion of phenol (C6H5OH) that corresponds to the peak that is located at m/z = 94. The phenol molecule that has been ionised without being fragmented is represented by this base peak, which is named after the phenol molecule. There is a correlation between the m/z value of this peak with the molecular weight of phenol, which is 94 grammes per mole.

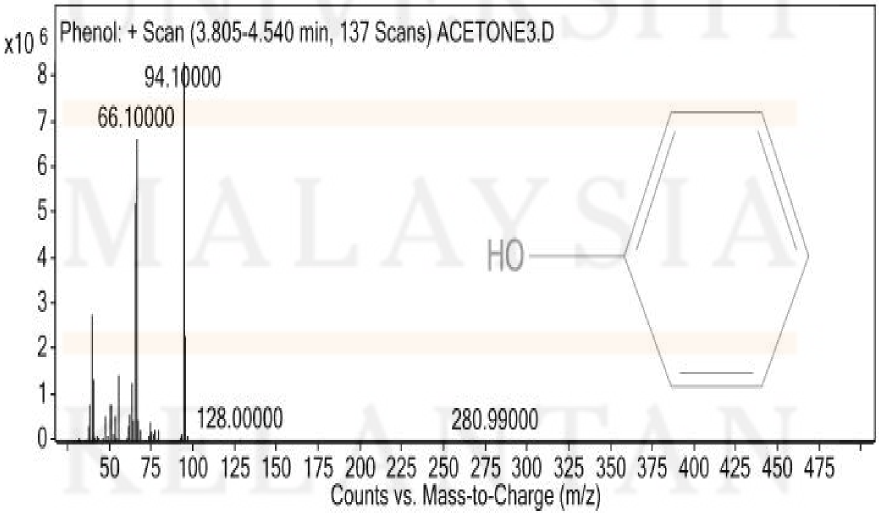

Figure 5.6: Shows mass-to-charge ratio (m/z) at 0.30 reaction.

There is a possibility that the peak at m/z 94 matches the molecular ion of phenol itself, which has a molecular weight of 94 grammes per molecular unit. What this indicates is that there may be some phenol in the sample that has not yet been reacted. The peak at m/z 66 might be a fragment of an oxidation product or a smaller organic component that could be produced during the oxidation process. Both of these possibilities are possible. It is more challenging to assign the peak at m/z 128; this is because more information is required. The molecular weight of this substance is greater than that of phenol, suggesting that it may be a dimer or an oxidation product.

The reaction efficiency and product profile may be assessed by GC-MS analysis, which is greatly affected by the catalyst mass.The oxidation process and catalyst selectivity may be better understood with the use of GC-MS analysis, which enables the identification and quantification of reaction products. To determine how catalyst loading affects reaction routes, scientists can alter the silica nanoparticle mass and see how the product distribution varies.Analysis using gas chromatography-mass spectrometry allows scientists to detect any possible side reactions or by-products and determine the effect of catalyst mass on selectivity towards target oxidation products. To optimise the reaction conditions and obtain the required phenol conversion and product profile, this information is critical.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Silica nanoparticles from rice husk ash (RHA) have been used to oxidise phenol due to their catalytic capability and sustainability. This technology uses silica nanoparticles to oxidise phenol, a typical industrial effluent contaminant, into less hazardous compounds. Due to their copious availability, low cost, and great surface area, rice husk ash silica nanoparticles are attractive catalytic platforms. Molecular oxygen activation generates reactive oxygen species that attack the phenolic ring to yield oxidised products.

A key factor in oxidation efficiency is catalyst silica nanoparticle mass. The catalyst's mass determines the amount of active sites, which impacts phenol conversion and reaction rate. Experimental studies have shown that catalyst mass affects phenol oxidation using rice husk ash silica nanoparticles. Researchers monitored reaction kinetics, selectivity for desired products, and catalyst stability while systematically varying catalyst mass. Careful investigation can find the best catalyst mass for phenol conversion, economic viability, and environmental sustainability.

A few analyses from the XRD, SEM, EDX, FTIR, TGA and DSC were used to characterise the formation of RHA. The impact of catalyst mass on phenol oxidation using silica nanoparticles derived from rice husk ash was examined using Energy Dispersive X-ray Spectroscopy (EDX) and Scanning Electron Microscopy (SEM). SEM revealed details on the shape of the catalyst, while EDX revealed its elemental makeup. The chemical make-up and shape of the silica nanoparticles, as well as their catalytic activity, were shown to be altered by changes in the catalyst mass, according to the results. The XRD diffractogram demonstrates the samples are in the amorphous form. In addition, the surface structure of the sample is shown in the SEM image with agglomerate, spherical particles, and some irregular, rock-like particles. FTIR analysis revealed changes in silica nanoparticles' functional groups with varying catalyst mass during phenol oxidation. Mass variation influenced Si-OH, C-O, and Si-O-Si bands, indicating mass-dependent structural alterations affecting catalytic activity. TGA and DSC were used to evaluate phenol oxidation using rice husk ash-derived silica nanoparticles. TGA showed weight loss profiles, showing organic compound degradation, whereas DSC showed heat flow fluctuations, indicating reaction kinetics. The oxidation procedure affected the catalyst's thermal behaviour, revealing its stability and reactivity.

A chemical product known as phenyl β-D-glucopyranoside is formed when a glucose molecule is connected to a phenyl group via a β-glycosidic bond. The chemical is a glycoside, more precisely a glucoside, with glucose as the sugar moiety and phenyl as the aglycone moiety. A simple sugar molecule found in nature. A substrate for enzymes and a marker in chromatographic tests are two of the many uses for this sugar molecule.Glycoside hydrolase enzymatic tests and organic synthesis both make frequent use of this molecule. Carbohydrate chemistry and the processes of glycoside bond cleavage may be studied using it as a model chemical.

5.2 Recommendation:

An attractive path for scientific inquiry is the usage of nanoparticles formed from rice husk ash for the oxidation of phenol. This is due to the potential ecological benefits and the efficient disposal of agricultural leftovers that may be achieved via this method.

1. The Comprehensive Assessment of the Environmental Impact:

Analyse the effects that the oxidation process has on the environment, taking into consideration factors such as the amount of energy used, the amount of waste produced, and the ecological balance of the catalyst. The phenol oxidation capabilities of nanoparticles produced from rice husk ash are compared to those of other catalysts in terms of environmental sustainability and efficiency. A comparison is made between the two.

2. The analysis of kinetics:

Making use of the relevant kinetic models, one must ascertain the kinetic parameters of the oxidation reaction. To understand the rate-determining stages and evaluate the catalytic efficiency of various nanoparticle catalysts, kinetic studies have to be conducted under a variety of situations.

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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