The Effect of Reaction Time on the Viscosity and Density of Tetraethyl Orthosilicate from Silica of Rice Husk Ash

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Received: *29 November 2023;* ***Accepted:*** *9 December 2023;* ***Published:*** *11 December 2023*

Abstract: TEOS is a material widely used in various industrial fields. One source of silica (SiO_2) is rice husk ash. The purpose of this study was to determine the effect of reaction time on viscosity and density in making TEOS from rice husk silica. Silica resulting from the purification of rice husk ash is used in the TEOS manufacturing process by examining the variation of reaction time. One mole of ethanol (58.4 ml) and 0.25 mole (7 gr) of silica powder were added to a 250 ml round bottom flask, followed by the addition of 1 gr of CuO/Al_2O_3 catalyst. The mixture was then refluxed for 30, 35, 40, 45, and 50 hours with effective stirring at a temperature of 90°C. The FTIR characterization results show that there are three main functional groups: the -OH, Si-O, and C-O groups in the five TEOS synthesis results. Wave numbers of the –OH functional groups obtained ranged from 3349 cm^{-1} to 3315 cm^{-1}; Si-O functional groups ranged from 813 cm^{-1} to 606 cm^{-1}, and C-O functional groups ranged from 1105 cm^{-1} to 1040 cm^{-1}. Reaction time has a significant effect on the viscosity and density of the resulting TEOS. The findings indicate that reaction time does not significantly affect the viscosity of TEOS solutions within the investigated time frame. The viscosity values obtained fall within the acceptable range for commercial TEOS, indicating successful production of TEOS solutions with suitable flow characteristics. However, the density values obtained do not meet the requirements for commercial TEOS, highlighting the need for further optimization.

Keywords: Rice husk, reaction time, viscosity, density, and TEOS

1. Introduction

TEOS is widely used by semiconductor factories in Indonesia. Its applications include the production of ceramics, corrosion-resistant coatings, semiconductor devices, composite base materials (Dasmawati et al., 2011), heterogeneous catalysts (Fatimah et al., 2008), electronic substrates, thin-film substrates, adsorbents (Suarya et al., 2010), and electrical insulators (Zawrah et al., 2009; Alhussein et al., 2016). The advantage of using TEOS as a source or precursor of silica lies in its production of very fine silica particles (Alhussein et al., 2016). So far, TEOS has been imported from China and Japan. On the other hand, silica is

abundantly found in nature, such as in sand, quartz, glass, rice husk, and so on (Sulastri & Kristianingrum, 2010; Soltani et al., 2015).

Rice husk is an agricultural residue that is abundant in Indonesia, including in the South Kalimantan province. The Central Statistics Agency (BPS) recorded rice production in South Kalimantan in 2015 reaching 2.14 million tons, an increase of 45 thousand tons or 2.18% compared to the 2014 production of 2.09 million tons. Rice husk ash contains silica ranging from 87% to 97% dry weight (Handayani et al., 2014), while a study conducted in 2010 by Mujiyanti et al. found that rice husk ash contains 95.6% silica. The high silica content in rice husk suggests its potential as a raw material for TEOS production. The synthesis of TEOS from silicon powder and ethanol using alumina oxide catalyst has been conducted by Alhussein et al. (2016) with a product yield of 80%. The reaction took place over 40 hours with an ethanol : silica molar ratio of 4:1. Therefore, in this study on TEOS production from rice husk, we followed the method used by Alhussein et al. (2016) with variations in reaction time. The resulting TEOS compound was analyzed for its physical properties, including viscosity, density, and functional groups, using Fourier Transform Infrared (FTIR).

While previous studies have examined the synthesis of tetraethyl orthosilicate (TEOS) from silica derived from rice husk ash, there is a research gap concerning the specific impact of reaction time on the viscosity and density of the resulting TEOS product. Although the synthesis process involves various factors like temperature, reactant concentration, and catalyst type, the influence of reaction time on the physical properties of TEOS has not been extensively investigated. Understanding the relationship between reaction time and the viscosity and density of TEOS is crucial for optimizing the synthesis process and ensuring desired product quality. Viscosity and density are significant parameters that directly affect the usability and applicability of TEOS in various industrial applications, such as sol-gel coatings, silicon-based materials, and optical fibers. By exploring the effect of reaction time on the viscosity and density of TEOS derived from rice husk ash silica, researchers can gain insights into the kinetics and molecular structure of the reaction. This knowledge can help determine the ideal reaction time necessary to achieve the desired viscosity and density of TEOS, leading to improved process efficiency and cost-effectiveness. Moreover, comprehending the impact of reaction time on the physical properties of TEOS can contribute to the development of more sustainable and environmentally friendly processes. Rice husk ash, an abundant agricultural waste product, holds promise as a precursor for TEOS synthesis, offering opportunities for waste valorization and reducing dependence on traditional silica sources. Therefore, investigating the research gap concerning the effect of reaction time on the viscosity and density of TEOS derived from rice husk ash silica can provide valuable insights into optimizing the synthesis process, enhancing understanding of TEOS properties, and advancing sustainable materials synthesis.

2. Methodology

2.1 Equipment and Materials

The equipment used in this study includes a hot plate stirrer (Stuart), analytical balance (OHAUS), Muffle furnace, 240-mesh sieve, glassware (dropper pipet, volumetric pipet, Erlenmeyer flask, beaker, funnel, round-bottom flask), Fourier Transform Infrared Spectroscopy (Shimadzu Prestige 21), oven (Memmert), porcelain crucible, pycnometer, viscometer. The materials used in this study are rice husk, 95-97% ethanol (MERCK), 37% HCl (MERCK), Al_2O_3, Whatman No. 42 filter paper, and distilled water.

2.2 Rice Husk Ash Preparation

The husks are washed with water to remove impurities, especially clay, and then dried under sunlight. The dried husks are weighed to obtain a 50 gr sample. The sample is then incinerated at a temperature of 600ºC for 4 hours. The high-temperature heating process is carried out to eliminate organic components using a heating furnace. The rice husk ash is ground and sieved with a 240-mesh sieve to achieve a homogeneous size (Ginanjar et al., 2015).

2.3 Purification of Rice Husk Silica

The purification process involves placing the sample, in the form of rice husk ash, into a beaker, moistening it with hot distilled water, and then adding 5 ml of concentrated HCl before evaporating it to dryness. The addition of HCl is performed three times. Once the sample is dry, 20 ml of distilled water and 1 ml of concentrated HCl are added, and it is left on a water bath for 30 minutes. The sample is then filtered using ash-free filter paper and washed 4 to 5 times with hot distilled water. The result of the filtration, comprising solid residue along with the filter paper, is first heated at 300ºC for 3 hours until the filter paper turns into charcoal. This is followed by further heating at 600ºC until only white silica (SiO_2) sediment remains (Mujiyanti et al., 2010).

2.4 Synthesis of CuO/Al_2O_3 Catalyst by Impregnation Method

The γ-Al_2O_3 support is activated in an oven for 2 hours with 9 grams at 110°C. An impregnation solution is prepared by dissolving 9 grams of γ-Al_2O_3 in 30 ml of distilled water in a 250 ml beaker and stirred (Solution I). Additionally, 2.683 grams of Copper (II) Chloride dihydrate (CuCl2.2H2O) are weighed and dissolved in 70 ml of distilled water in a 100 ml beaker and stirred (Solution II). Subsequently, Solutions I and II are combined in another beaker and heated to a temperature of 60-70°C while stirring until a dry or pasty mixture is formed. The formed paste is then dried in an oven at 120°C for 2 hours. Finally, it is calcined in a furnace at 500°C for 5 hours.

2.5 TEOS Synthesis Process

A total of 1 mole of ethanol (58.4 ml) and 0.25 mole (7 grams) of silica powder were added to a 250 ml round-bottom flask, followed by the addition of 1 gr of alumina oxide catalyst. The mixture was then refluxed for varied reaction times of 30, 35, 40, 45, and 50 hours, with effective stirring and a temperature of 90°C. The reaction was monitored until the hydrogen gas formation reached a stable state. At the end of the reaction, the mixture was filtered to separate silicon and the catalyst. The produced TEOS underwent viscosity and density tests and was further analyzed using FTIR (Alhussein et al., 2016).

2.6 Data Analysis

Data analysis was conducted by presenting the data obtained from this research, including viscosity, density, and infrared spectrum data, in the form of tables or graphs. The data were then analyzed descriptively to observe the influence of reaction time on TEOS formation by comparing it with references from previous studies. Viscosity and density data were compared with the reference values for commercial TEOS.

3. Result and Discussion

3.1 Rice Husk Ash Preparation

This preparation stage is conducted to eliminate organic components such as cellulose, hemicellulose, and lignin present in rice husk (Chandra et al., 2012; Ginanjar et al., 2015). The preparation process begins with washing the rice husk to remove impurities such as mud, dust, and others. The washed rice husk is then dried under sunlight until it is completely dry. Incineration is carried out at a temperature of 600°C for 4 hours to eliminate organic compounds in the rice husk and prevent the transformation of amorphous silica into crystalline form (Chandra et al., 2012). The result of the rice husk incineration is white to grayish ash, which is then sieved using a 240-mesh sieve to standardize particle size for more effective purification in the next stage (Sari, 2013). The obtained rice husk ash content is 24.45%. In a previous study by Nopianingsih et al. (2015), incinerating 1000 g of rice husk in a furnace at 700°C resulted in an ash content of 20.20%, appearing white. Chakraverty et al. (1988) analyzed rice husks heated in a controlled combustion furnace with temperatures ranging from 300°C to 700°C to compare the color of the resulting ash. The results indicated that the lower the combustion temperature, the longer it took to produce whitish rice husk ash. This is because the combustion of carbon in rice husk at low temperatures has a slower burning rate (Chakraverty et al., 1988).

3.2 Purification of Rice Husk Silica

The silica content of purified rice husk ash is 87.87%. According to a previous study conducted by Maulidiyah (2017), purified rice husk ash, characterized by XRF using a catalyst, yielded a silica (Si) content of 89%. Maulidiyah (2017) also reported that purifying rice husk ash using HCl can enhance the percentage of Si content in rice husk ash.

3.3 CuO/Al_2O_3 Catalyst Preparation by Impregnation Method

The CuO/Al_2O_3 catalyst was prepared using the impregnation method, beginning with the initial drying of the γ-Al_2O_3 carrier in an oven for 2 hours to evaporate impurities that may hinder the penetration of the impregnation solution into the carrier material (Dewi et al., 2016). Subsequently, γ-Al_2O_3 was dissolved in 30 ml of distilled water (Solution I), and copper (II) chloride dihydrate (CuCl2.2H2O) was dissolved in 70 ml of distilled water (Solution II) and stirred until fully dissolved. Solutions I and II were then mixed in another beaker and heated to a temperature of 60-70°C while stirring until a dry or pasty consistency formed. Once the paste was formed, it underwent drying in an oven at 120°C for 2 hours. The drying process aimed to crystallize metal salts on the surface of the carrier's pores. If not done correctly, an uneven concentration distribution may result (Dewi et al., 2016). Finally, it was calcined in a furnace at 500°C for 5 hours. Calcination is a high-temperature heating process, still below the melting point, designed to remove solvents. The calcination process of the impregnation result causes the release of water, increasing the outer surface area of the catalyst's pores, thereby enhancing absorption capacity (Dewi et al., 2016).

3.4 TEOS Synthesis Process

TEOS synthesis was conducted with variations in reaction time: 30, 35, 40, 45, and 50 hours, involving the mixing of 1 mole of ethanol (58.4 ml), 0.25 moles (7 grams) of silica powder, and 1 gram of CuO/Al_2O_3 catalyst. The mixture was then refluxed at a temperature of 90°C. The addition of the catalyst aims to expedite the reaction process. The resulting reflux

was filtered to separate the solid and liquid components. The obtained filtrate is a slightly cloudy, transparent liquid with an alcohol-like odor, resembling the properties of TEOS (NIOSH, 2005). The volumes for each variation in reaction time were 30 ml, 39 ml, 27 ml, 22 ml, and 18 ml, respectively.

3.5 TEOS Synthesis Process

Functional group analysis was conducted to identify the functional groups present in the refluxed solution. The compounds resulting from reflux were analyzed in the wavenumber range of 400-4500 cm^{-1}. The FTIR spectra of the compounds resulting from reflux with varying reaction times can be seen in Figure 2.

Based on the FTIR spectrum in Figure 2, it can be observed that there is no significant change in transmittance for the five samples. The FTIR measurements revealed several functional groups with specific wavenumbers in these five samples, as shown in Table 1. The Si-O functional group can be observed in each variation over time, with differences in wavenumbers for both symmetric and asymmetric Si-O. In variation A30 (30 hours), the Si-O asymmetric functional group was observed with an absorption band at 813 cm^{-1}. For sample A35 (35 hours), there was a shift in the wavenumber to 800 cm^{-1}, sample A40 (40 hours) showed a further shift to 798 cm^{-1}, and then in sample A45 (45 hours), the Si-O wavenumber shifted back to 800 cm^{-1}, like sample A35. In sample A50 (50 hours), the wavenumber of the Si-O asymmetric group was the same as in sample A40, which is 798 cm^{-1}.

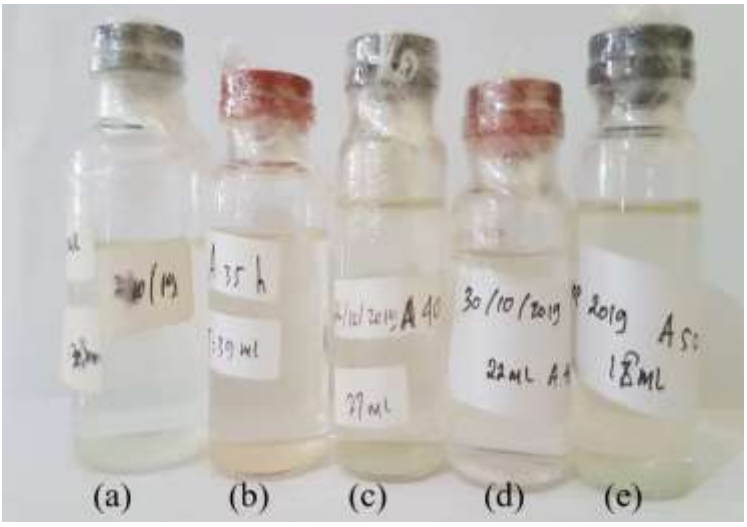

Figure 1. Reflux Results a) 30 hours; b) 35 hours; c) 40 hours; d) 45 hours; and e) 50 hours

The Si-O symmetric functional group in A30 had the same wavenumber as A45, which is 640 cm^{-1}. However, for other variations, the wavenumbers were different. The Si-O symmetric functional group in A35, 40, and 50 were indicated at wavenumbers 650 cm^{-1}, 616 cm^{-1}, and 606 cm^{-1}, respectively.

Additionally, the C-O functional group showed different absorption bands in each time variation. For sample A30, the wavenumbers were 1104 cm^{-1} and 1050 cm^{-1}; A35 had 1105 cm^{-1} and 1040 cm^{-1}; A50 had 1105 cm^{-1} and 1056 cm^{-1}; A40 and A45 had wavenumbers for the C-O group at 1098 cm^{-1}, 1050 cm^{-1}, 1095 cm^{-1}, and 1056 cm^{-1}, respectively. These differences in wavenumbers among the five samples indicate the influence of reaction time and interactions with other related functional groups (Pavia et al., 2015).

The C-H functional groups in the five samples also experienced a shift in wavenumbers. The shifts in the wavenumbers of the symmetric and asymmetric stretching C-H groups were not significant. For the symmetric C-H, there was a shift towards higher wavenumbers, but in sample A50, the wavenumber shifted back towards lower values. The asymmetric C-H groups

experienced fluctuating shifts, with values for A30 at 2967, 2888 cm^{-1}; A35 at 2980, 2895 cm^{-1}; A40 at 2975, 2887 cm-1; A45 at 2970, 2892 cm^{-1}; and A50 at 2960, 2895 cm^{-1}. This absorption band data indicates the presence of C-H groups in each sample. These C-H groups may originate from TEOS, such as Si(OC2H5)4, or possibly from ethanol.

The O-H functional groups are evident in all five samples, both in stretching and bending. From the table, absorption bands for the stretching of O-H groups were obtained for each sample: 3349 cm^{-1}, 3315 cm^{-1}, 3344 cm^{-1}, 3325 cm^{-1}, and 3315 cm^{-1}. Samples A35 and A50 have the same wavenumber for O-H. These -OH functional groups can be assumed to originate from ethanol still present in the samples, as indicated by the research conducted by Alhussein et al. (2016), which found that analysis using Gas Liquid Chromatogram (GLC) revealed the presence of around 20% ethanol. Based on the results of the previous study by Alhussein et al. (2016) presented in Figure 3 and Table 3, a comparison with the current study's data indicates that the spectra of the five analyzed samples are nearly similar.

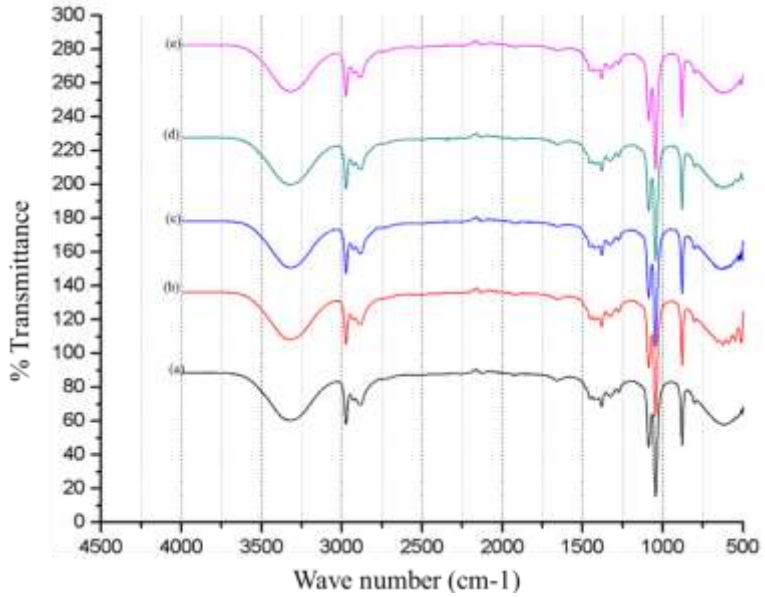

Figure 2. IR Spectrum of reflux results a) 30 hours, b) 35 hours, c) 40 hours, d) 45 hours, and e) 50 hours

3.6 Determination of Viscosity and Density

The determination of viscosity is a crucial aspect in assessing the flow rate of a solution (Purwanti et al., 2022). In Table 2, the viscosity results of the reaction solution are presented. The data reveals that at 30, 35, and 45 hours, the viscosity values are relatively similar, measuring 3.67 cps, 3.21 cps, and 3.16 cps, respectively. On the other hand, the samples at 40 and 50 hours exhibit lower viscosities, measuring 2.90 cps and 2.74 cps, respectively. It is worth noting that the viscosity values obtained from Table 2 fall within the standard range for commercial TEOS, which typically ranges from 1-3 cps.

Moving on to density determination, the objective is to ascertain the mass density of a solution or compound. The density results from the reaction solution are presented in Table 3. The purpose of including various reaction times (30, 35, 40, 45, and 50 hours) in Table 3 is to investigate whether there is an influence on density (Gaina et al., 2023). Upon examination, it can be observed that the mass density for each treatment does not exhibit significant differences. However, it is important to note that the obtained results do not yet meet the density values required for commercial TEOS.

The viscosity and density measurements play a crucial role in evaluating the quality and suitability of TEOS for different applications. Viscosity is a key parameter that determines the flow characteristics and processability of TEOS-based solutions. The obtained viscosity values

indicate that the reaction process has produced TEOS solutions with viscosities within the acceptable range for commercial applications. This suggests that the reaction time does not significantly affect the viscosity of the TEOS solution, at least within the investigated time frame.

Density, on the other hand, provides insights into the compactness and mass per unit volume of the TEOS solution. While the density values obtained from the reaction process do not meet the requirements for commercial TEOS, further investigation is needed to understand the factors influencing density and to optimize the reaction conditions accordingly. It is important to consider that the determination of viscosity and density alone may not provide a comprehensive understanding of the overall quality and performance of TEOS derived from rice husk ash silica. Additional characterization techniques, such as spectroscopic analysis or mechanical testing, may be necessary to evaluate other relevant properties, including chemical composition, structural integrity, and mechanical strength. Future research endeavors could focus on exploring the relationship between reaction time and other physical and chemical properties of TEOS derived from rice husk ash silica. Investigating the influence of reaction time on parameters such as surface tension, refractive index, or chemical reactivity could provide a more comprehensive understanding of the synthesis process and its impact on the final TEOS product.

The presented data highlights the viscosity and density results obtained from the reaction process of TEOS derived from rice husk ash silica. The viscosity values fall within the acceptable range for commercial TEOS, indicating that the reaction process has yielded solutions with suitable flow characteristics. However, the density values obtained do not meet the requirements for commercial TEOS, suggesting the need for further optimization. Exploring the relationship between reaction time and other properties of TEOS can contribute to a deeper understanding of the synthesis process and aid in the development of high-quality TEOS materials.

Table 1. IR Spectrum peak identification

Wave number (cm^{-1})					Vibration Type
A30	A35	A40	A45	A50	
3349	3315	3344	3325	3315	O-H strain
2967	2980	2975	2970	2960	C-H asymmetric strain
2888	2895	2887	2892	2895	
2615	2625	2709	2750	2744	C-H symmetric strain
1650	1656	1670	1648	1665	O-H bend
1465	1470	1458	1470	1480	
1393	1400	1396	1389	1351	C-H bend
1332	1325	1336	1335	1328	
1278	1260	1280	1287	1290	CH_3 wobble
1104	1105	1098	1095	1105	C-O strain
1050	1040	1050	1056	1056	
870	884	875	890	884	C-C strain
813	800	798	800	798	SiO_4 asymmetric strain
640	650	616	640	606	SiO_4 symmetric strain

Table 2. Results of viscosity determination from the reaction solution

Sample	V	TEOS viscosity value (Zhangjiagang Fortune Chemical Co., Ltd)
A30	3.67	4-7 cps (grade-40),
A35	3.21	1-3 cps (grade-32), dan
A40	2.90	0.97 cps (grade-28)

A45	3.16	
A50	2.74	

Table 3. Results of density determination from the reaction solution

Sample	V	TEOS viscosity value (Zhangjiagang Fortune Chemical Co., Ltd)
A30	0.7917	
A35	0.8101	1.05-1.07 g/ml (grade-40)
A40	0.8144	0.97-1.00 g/ml (grade-32)
A45	0.8204	0.93-0.94 g/ml (grade-28)
A50	0.8131	

4. Conclusions

The effect of reaction time on the viscosity and density of tetraethyl orthosilicate (TEOS) derived from silica of rice husk ash has been investigated, shedding light on several important aspects. The research findings indicate that reaction time does not significantly impact the viscosity of TEOS solutions within the investigated time frame. The viscosity values obtained fall within the acceptable range for commercial TEOS, suggesting that the reaction process has successfully produced TEOS solutions with suitable flow characteristics. On the other hand, the density values obtained from the reaction process do not meet the requirements for commercial TEOS. This indicates that further optimization is necessary to achieve the desired mass density. While the research has identified a research gap in understanding the specific influence of reaction time on the viscosity and density of TEOS derived from rice husk ash silica, it also highlights the need for additional investigations to determine the factors affecting density and to optimize the reaction conditions accordingly. The research on the effect of reaction time on the viscosity and density of TEOS from rice husk ash silica has practical implications for various industries.

Understanding the relationship between reaction time and the physical properties of TEOS is crucial for optimizing the synthesis process and ensuring the desired product quality. Controlling viscosity and density within specific ranges is essential to meet the required specifications for different applications, such as sol-gel coatings, silicon-based materials, and optical fibers. Moreover, the utilization of rice husk ash as a precursor for TEOS synthesis offers environmental benefits by repurposing agricultural waste. Investigating the effect of reaction time on the viscosity and density of TEOS derived from rice husk ash silica aligns with the principles of sustainable materials synthesis. It contributes to the development of eco-friendly processes that reduce reliance on traditional silica sources and promote waste valorization, thus minimizing the environmental footprint of TEOS production. While the research has provided valuable insights into the viscosity and density of TEOS, it is important to consider that these properties alone may not provide a comprehensive understanding of the overall quality and performance of TEOS derived from rice husk ash silica. Further characterization techniques and investigations into other physical and chemical properties, such as surface tension, refractive index, or chemical reactivity, may be necessary to evaluate the complete set of relevant properties.

The research on the effect of reaction time on the viscosity and density of TEOS derived from silica of rice husk ash has contributed to the optimization of the synthesis process, understanding of TEOS properties, and advancement of sustainable materials synthesis. Further research endeavors can build upon these findings to explore the relationship between reaction time and other properties of TEOS, leading to the development of high-quality TEOS materials with enhanced performance for a wide range of industrial applications.

Acknowledgements

The author expresses gratitude to the Inorganic Chemistry Laboratory of the Faculty of Mathematics and Natural Sciences, Lambung Mangkurat University, for providing facilities for the implementation of this research.

References

Alhussein, O., Elgorashi, E., & Bayahia, H. (2016). A Novel Method for Synthesis of Ethylsilicates and its Applications. *Asian Journal of Chemistry*, *28*(12), 2741-2744.

Chakraverty, A., Mishra, P., & Banerjee, H. D. (1988). Investigation of combustion of raw and acid-leached rice husk for production of pure amorphous white silica. *Journal of Materials Science*, *23*, 21-24.

Chandra, A., Miryanti, Y. I. P., Widjaja, L. B., & Pramudita, A. (2012). Isolasi dan karakterisasi silika dari sekam padi.

Dewi, T. K., Mahdi, M., & Novriyansyah, T. (2016). Pengaruh rasio reaktan pada impregnasi dan suhu reduksi terhadap karakter katalis kobalt/zeolit alam aktif. *Jurnal Teknik Kimia*, *22*(3), 9-18.

Fatimah, I., Rubiyanto, D., & Huda, T. (2008). Peranan katalis TiO_2/SiO_2-montmorillonit pada reaksi konversi sitronelal menjadi isopulegol. *Reaktor*, *12*(2), 83-89.

Gaina, P. C., Sari, M. M., Suryawan, I. W. K., Prayogo, W., Ummatin, K. K., Arifianti, Q. A. M. O., & Faria, N. (2023). Characteristics of Density and Hardness on Caloric Value of Substitution of Biomass and Pet Plastics as Refused Derived Fuel Pellets. *Jurnal Bahan Alam Terbarukan*, *12*(1), 39-44.

Ginanjar, R. R. (2015). *Ekstraksi Silika Dari Abu Sekam Padi Menggunakan Pelarut NaOH* (Doctoral dissertation, Universitas Muhammadiyah Purwokerto).

Handayani, P. A., Nurjanah, E., & Rengga, W. D. P. (2014). Pemanfaatan limbah sekam padi menjadi silika gel. *Jurnal bahan alam terbarukan*, *3*(2), 55-59.

Maulidiyah, A. N. (2017). *Sintesis dan karakterisasi nanozeolit Y dari abu sekam padi dengan variasi suhu hidrotermal* (Doctoral dissertation, Universitas Islam Negeri Maulana Malik Ibrahim).

Mujiyanti, D. R., Astuti, M. D., & Umaningrum, D. (2010). Pembutan Silika Amorf pada Limbah Sekam Padi Gambut di Kabupaten Banjar Kalimantan Selatan. *Laporan Penelitian*.

NIOSH. (2005). NIOSH pocket guide to chemical hazards and other databases CD-ROM. Department of health & human services, centers for disease prevention & control.

Nopianingsih, N. N. S., Sudiarta, I. W., & Sulihingtyas, W. D. (2015). Sintesis silika gel terimobilisasi difenilkarbazon dari abu sekam padi melalui teknik sol gel. *Jurnal Kimia*, *9*(2), 226-234.

Purwanti, A., Yusuf, M., Prayogo, W., & Ambarita, U. A. (2022). Penentuan koefisien transfer massa padat cair pada sistem tawas air dengan variasi kecepatan putaran dan diameter pengaduk. *KURVATEK*, *7*(1), 63-70.

Sari, D. M. 2013. Uji Fisis Komposit Polimer Resin Epoksi Berbasis Abu Sekam Padi dengan Agregat Pasir Sungai. Skripsi Jurusan Fisika, Universitas Lampung, Bandar Lampung.

Soltani, N., Bahrami, A., Pech-Canul, M. I., & González, L. A. (2015). Review on the physicochemical treatments of rice husk for production of advanced materials. *Chemical engineering journal*, *264*, 899-935.

Suarya, P., Putra, A. B., & Wisudawan, D. (2010). Interkalasi tetraetil ortosilikat (teos) pada Lempung teraktifasi asam sulfat dan pemanfaatannya Sebagai adsorben warna limbah garmen. *J. Kimia*, *4*(1), 43-48.

Sulastri, S., & Kristianingrum, S. (2010). Berbagai macam senyawa silika: Sintesis,

karakterisasi dan pemanfaatan. In *Prosiding Seminar Nasional Penelitian, Pendidikan dan Penerapan MIPA* (pp. 211-216).

Zawrah, M. F., El-Kheshen, A. A., & Abd-El-Aal, H. M. (2009). Facile and economic synthesis of silica nanoparticles. *Journal of Ovonic Research*, *5*(5), 129-133.