

Effect of Chemical Treatments on Rice Husk (RH) Water Absorption Property

Zeynab Emdadi, Nilofar Asim, M. Ambar Yarmo, and K. Sopian

Abstract—This study involves the investigation of replacing chemical desiccants with natural fibers as a novel and sustainable alternative materials. In this study, the feasibility of using rice husk (RH) and its treated forms with alkali and acid solution and their blends as desiccant materials have been investigated. The bare RH, treated RH and the mixed materials have been characterized using FTIR, XRD, TGA, BET and SEM. The ability of water absorbance for the prepared samples has been compared at 28 ± 2 °C. The elementary test of water absorbance was determined to be in the following order: acid treated RH > alkali treated RH > bare RH. The composite samples with higher percentage of acid treated RH showed higher water absorbance which is likely to be attributed to the porosity and improvement of adhesion properties (surface hydroxyl groups) in the prepared samples.

Index Terms—Chemical treatments, rice husk (RH), water absorption.

I. INTRODUCTION

Desiccants act is considered as moisture absorber in order to dry their surrounding environments. The different water vapour pressure between the surrounding air and the desiccant surface is the reason for absorbing or adsorbing water vapour in desiccant materials. Drying mitigates the hazardous side effects of moisture, such as corrosion, swelling, and the development of rust, mold, mildew, and fungus. Several materials have been utilized as desiccants to absorb different types of liquid. Most commonly used desiccant materials include lithium chloride, triethylene glycol, silica gels, aluminium silicates (zeolites or molecular sieves), aluminium oxides, lithium bromide solution and lithium chloride solution [1]. The majority of these materials are expensive, encouraging researchers to identify new and economic materials [2]-[5]. Various researchers have also studied the environmental impact of these desiccants [3]. Replacing chemical desiccants, including silica gel and molecular sieves, with natural fibres is a novel and sustainable alternative. High-fibre agricultural wastes are potential candidates as desiccants for using in desiccant-dehumidification. Meanwhile the utilization of agricultural solid wastes is proposed as a viable solution to reduce pollution while deriving economic benefits. Ziegler *et al.* proposed the use of grains as desiccants in a solar-drying

storage system, utilizing the deep-bed drying model [2]. Williams *et al.* [3] investigated the use of kenaf core as a desiccant, and compared it with silica gel to determine the suitability of the former as a packaging desiccant. Starch was determined to be suitable desiccant material for industrial, commercial, and residential applications [5]. The same researchers studied the use of coconut coir as a desiccant in an engineering process [6]. There is a large amount of agricultural waste, including rice husk (RH), coconut coir, which can be effectively utilized for a sustainable approach. In comparison with other biomasses, RH contains uncommon amount of silica. The modification of physical and chemical surface properties could increase the adhesion properties of rice husks. Chemical treatments consider as one of most used methods for the surface treatments of agricultural fibres. The most common chemical treatment method is acid and alkali treatment. They could improve the adhesion properties of treated samples. Therefore, this research investigates the possibility of using rice husk and its treated form and their blend as a desiccant material.

II. EXPERIMENTAL PROCEDURE

A. Alkali Treated Rice Husk by 1.0% (W/V) KOH

Rice husk was gathered from a local rice mill during milling season. First of all the rice husk was washed with deionized water and dried in an oven at 105 °C for 24 hours. The clean and dried rice husks were treated by soaking it in 1.0% (W/V) KOH solution at 100 °C for 3 hr (50g rice husk in 500mL alkali solution). It was then left overnight to dry, and later filtered. The filtrated rice husks (first) were washed by HCl 0.1% (W/V) solution and later washed with plenty of distilled water until it was neutralized (pH = 7). The precipitate was then dried in the oven at 105 °C for 12 hours [7].

B. Acid Treated Rice Husk by Using HNO₃ 8N

The clean and dried rice husks as mentioned in section 2.1 were selected as a precursor material for preparation of sample. Acid treatment was performed by boiling of rice husk at 80-85 °C for 4 hr with acid/rice husk ratio 10mL:1gr in HNO₃ 8N solution. Later, the treated rice husk were washed by NaOH 0.5% (W/V) solution and later washed with plenty of distilled water until it was neutralized (pH = 7). Finally, the precipitate was dried in the oven at 105 °C for 12 hours.

C. Preparation of Some Composites Using Treated Rice Husk and Raw Rice Husk

Regarding to initial test result that showed higher water absorption for acid treated RH compared to base treated RH,

Manuscript received September 5, 2014; revised November 23, 2014.

Zeynab Emdadi, Nilofar Asim, and K. Sopian are with the Solar Energy Research Institute, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia (e-mail: emdadi58@gmail.com, nilofarasim@ukm.edu.my, ksopian@vlsi.eng.ukm.my).

M. Ambar Yarmo is with School of Chemical Science & Food Technology, Faculty of Science and Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia (e-mail: ambar@pkisc.cc.ukm.my).

it was chosen for further study and composite preparation. Different composites have been prepared using acid treated RH, raw RH and polyvinyl alcohol (PVA).

For preparation of composites, first the treated RH and raw RH manually mix very well in dry form for 5 min. later the Polyvinyl alcohol (PVA) powder (5 weight% of total dry materials weight which employed as adhesive material) was added into the prepared treated RH and raw RH mixture. Then deionized water added to above mixture and mixed for another 10 min till gave homogen mixture.

Consequently, the mixture was poured into molds ($20 \times 20 \times 20$ mm³). The specimens were cured at room temperature for 24 hours, after removing from the mold they dried in the oven at 105 °C temperature for 2 hours and cooled in desiccator. The employment of PVA as binder (adhesive) not only provide the proper physical shape for characterization and application but also its low percentage in blend (5%) make the blend more environmentally friendly.

III. RESULTS AND DISCUSSION

The crystallinity analyses of the samples were conducted using Bruker DB-Advance X-ray Diffractometer (XRD), Germany (Fig. 1). Both treated RH show the crystalline silicon oxide phase at about $22.23^\circ 2\theta$ in the tetragonal system (JCPDF file no.01-082-1554). The result shows that the peak from semi crystalline carbon structure which was appeared in base treated RH has almost been removed by HNO₃ treatment.

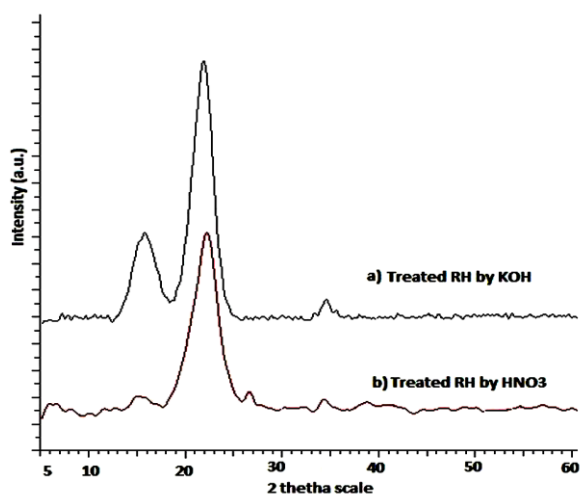


Fig. 1. XRD patterns of treated RH.

Fig. 2 shows the scanning electron micrographs of the surfaces of untreated rice husks and treated RH. The results showed that the surface of untreated rice husks is quite rough. The roughness of the surface is represented by the asperities (Fig. 2(a)). However, the asperities were almost removed from the surface after treatment in HNO₃ 8N solutions (Fig. 2(c)). Although, it seems that the acid treatment of rice husk can destroy the inherent structure of the husk.

The functional groups of the samples were determined using FTIR spectra (Fig. 3). The bands at 1023 cm⁻¹ and 792 cm⁻¹ are related to O-Si-O stretching vibrations. The treated RH show the band at 1635 cm⁻¹ which belongs to the C=O group and the band at 3339 cm⁻¹ is due to the chemisorbed water and surface hydroxyl groups. These peaks are

attributed to chemical treatment result for making more adhesive surface on treated RH.



Fig. 2. FESEM photographs of a) bare RH b) alkali treated RH and c) acid treated RH.

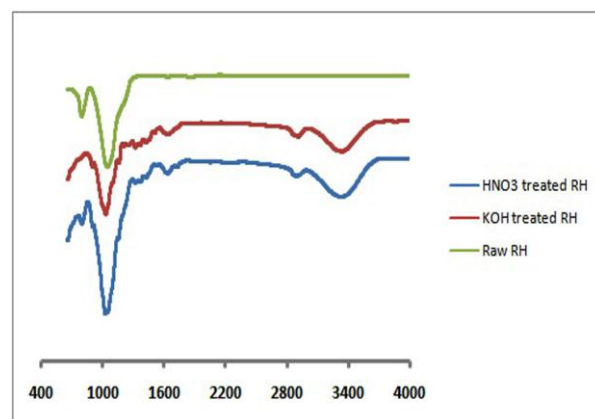


Fig. 3. FTIR spectra for raw RH and its treated forms.

The thermal stability of the treated RH was investigated and the results are shown in Fig. 4. The weight loss up to 110 °C is due to the removal of water. Most of the thermal degradation takes place in between 240 °C - 385 °C. In this area decomposition of primary hemicelluloses and cellulose takes place[8]. The decomposition of lignin compounds starts around 385 °C and terminates at around 850 °C. This reveals the influence of acid treatment on thermal stability of the rice

husks. The result showed that acid treated RH has improved thermal stability compared to base treated RH.

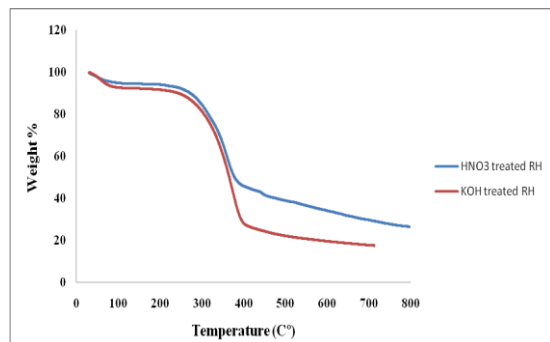


Fig. 4. TGA of HNO₃ treated rice husk and KOH treated rice husk.

The water absorption property of prepared samples has been examined for evaluation of desiccant performance. The ASTM D 570 – 98 has been used for measurement. The chemical structure and hydroxyl groups in the chain of cellulosic material are the reason for their absorbing water property [9]. Table I demonstrates the water absorption results for prepared samples and their compositions. The temperature has been chosen at 28 ± 2 °C. The elementary test of water absorbance was determined to be in the following order: acid treated RH > alkali treated RH > bare RH. The detailed measurement has been conducted for prepared blends. As can be seen, the absorption of water rises gradually during the time for blend samples. The results show higher water absorbance for blend with higher percentage of acid treated RH in their composite. One reason for this behaviour could be the higher percentage of hydroxyl group on treated RH surface after acid treatment. The sample A showed highest water absorption among all blend samples.

TABLE I: THE COMPOSITION OF PREPARED BLEND SAMPLE USING RAW RH, ACID BASE RH AND PVA AND THEIR WATER ABSORPTION

% Contents of materials				% Water absorption				
Sample	Treated RH by HNO ₃	Raw RH	PVA	After 10 minutes	After 30 minutes	After 1 hour	After 2 hours	After 24 hours
A	85	10	5	284.0	286.3	300.8	302.3	306.0
B	80	10	10	207.2	219.5	221.2	224.3	234.8
C	75	10	15	256.7	259.7	262.7	263.9	270.2
A'	80	15	5	276.1	283.2	285.7	290.2	295.6
B'	75	15	10	257.9	263.7	266.8	269.0	271.5
C'	70	15	15	253.8	255.0	266.0	267.9	270.8
A''	75	20	5	252.8	254.0	265.3	267.1	272.1
B''	70	20	10	209.5	212.6	219.5	221.8	223.9
C''	65	20	15	221.6	224.4	235.0	235.0	237.0
D	95	-	5	330.55	332.20	332.89	344.55	357.93
E	-	95	5	150.49	150.98	151.10	158.79	-

The specific surface area, pore size, pore volume, and pore diameter of the samples were determined by Brunauer-Emmet-Teller (BET) method [10] using a nitrogen adsorption instrument (Micrometrics ASAP 2010).

The results are listed in Table II. The results show higher surface area for sample A compared to A' and A''. This higher

surface area is another reason for higher water absorption result for sample A.

TABLE II: TEXTURE PROPERTIES OF SOME PREPARED SAMPLE

Physical properties	Samples			
	A	A'	A''	HNO ₃ treated RH
Surface area (m ² /g)	4.0879	2.8139	3.3662	9.1046
Pore size (nm)	21.8	19.0	23.2	16.2
Micropore volume (cm ³ /g)	-0.001504	-0.000990	-0.001490	-0.001117

The thermal stability of sample A, A' and A'' was investigated and the results are shown in Fig. 5. The weight loss up to 110 °C is due to the removal of water. Most of the thermal degradation takes place around 200 °C - 400 °C that is related to decomposition of primary hemicelluloses and cellulose. The next zone starts around 400 °C and terminates at around 800 °C is due to the decomposition of lignin compounds. The results show the higher thermal stability for sample A compared to samples A' and A''. The lignin has the highest thermal stability among the three components (cellulose, lignin, and hemicelluloses) and degrades at higher temperatures [11]. Acid treatment eliminates the cellulose and hemicelluloses more than lignin then the treated sample contain more lignin compared to raw sample. Therefore the higher thermal stability of A (with higher percentage of treated RH) compared to A' and A'' could attributed to higher percentage of lignin in its composition.

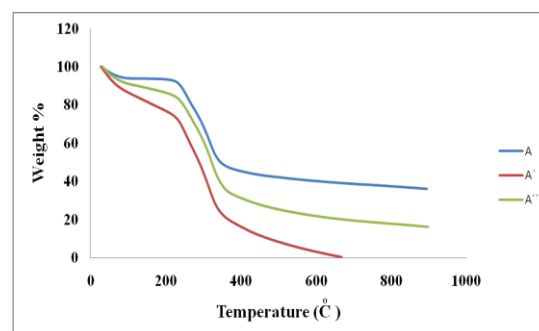


Fig. 5. TGA for samples A, A' and A''.

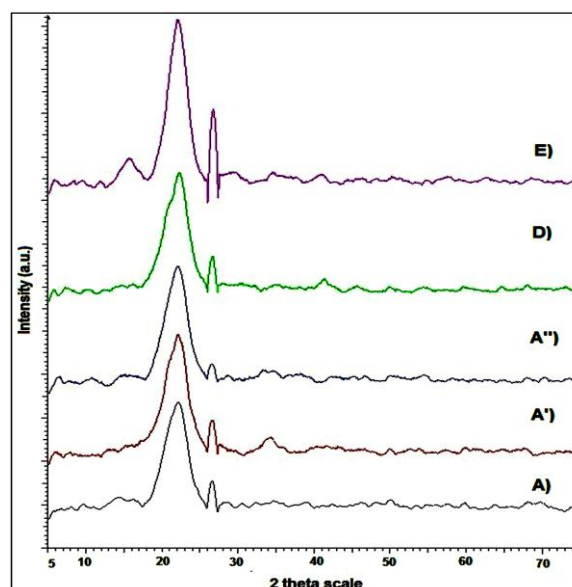


Fig. 6. XRD pattern of samples A, A', A'', D, E.

The X-ray diffraction (XRD) patterns for some of prepared blends are depicted in Fig. 6. All samples show crystalline SiO₂ phase pattern, although the intensity of it decrease by increasing the acid treated RH percentage in blend samples. The sample E which contain only raw RH and PVA shows semi crystalline peak for carbon from organic compounds that be reduce significantly by acid treatment.

IV. CONCLUSION

The initial findings in this research revealed that agricultural wastes have the potential to be used as a desiccant instead of chemical desiccants. The granular structure, insolubility in water, chemical stability, high mechanical strength and local availability at almost no cost of Rice husk (RH) make it a popular agricultural waste for investigation. The advantage in using these adsorbents in certain critical application is the fact that there is no need to regenerate them due to their low production costs. The chemical treatment influences the thermal properties, chemical structure and physical properties of rice husk. The result showed that treating the rice husk with a dilute solution of 1.0% KOH and 8M HNO₃ solution improve the bond strength of O-Si-O of the silica and reduces the quantity of amorphous silica. Besides, the treatment make more porous materials and create surface hydroxyl groups which increase the water absorption property.

The ability of water absorbance for the prepared samples has been investigated at 28 ± 2 °C. The results revealed the higher water absorbance of alkali treated RH, acid treated RH and their composites compared to bare RH. The composite samples with higher percentage of acid treated RH showed higher water absorbance. Although these finding are so promising for commercial application of treated RH, detailed studies involving the determination of the superior treatment condition and optimization of the procedure, investigating the life cycle of the rice husk (adsorption-regeneration), moisture absorption, and the quality of air leaving the bed must be conducted before totally commercial application.

ACKNOWLEDGEMENTS

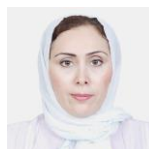
The authors would like to thank the DLP-2013-015 and GGPM-2012-027 Research Funds for providing financial support for this study.

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Zeynab Emdadi was born in Rasht, Iran in 1980. She obtained her BSc in in applied chemistry from Azad University of Iran in 2004, MSc in organic chemistry from K. N. Toosi University of Technology Department of Chemistry, Iran in 2008. She is currently a PhD student under the supervision of Assoc. Prof. Dr Nilofar Asim. Her current research focuses on study and evaluation of different porous geopolymers for green passive cooling application at Solar Energy Research Institute from Universiti Kebangsaan Malaysia.



Nilofar Asim was born in Tabriz, Iran in 1970. She obtained her BSc in in applied chemistry, MSc in inorganic chemistry from Tehran University (Iran) and PhD in materials science from National university of Malaysia. She is presently working at Solar Energy Research Institute, a center of excellence for the research and development in solar energy technology, Universiti Kebangsaan Malaysia. She has been involved in the field of materials science for more than 17 years.

Dr. Nilofar Asim's main contributions are in synthesis of nanomaterials (metal oxide and composite), photocatalysts, inorganic polymers and agriculture waste materials modification for new applications. She has published 50 research papers in journals and conferences. She is an IEEE member.



Mohd. Ambar Yarmo recieved his BSc in chemistry from Universiti Kebangsaan Malaysia (UKM) and his PhD in analytical chemistry from University Of Wales, Cardiff, U.K. Prof. Mohd. Ambar Yarmo's research interests are in conversion of CO₂ to Fuel, bio-ethanol derivatives and biofuel applications, upgrading of natural gas and palm oil to higher added value speciality chemicals using combinatorial technologies and catalysis. He has attended to Japanese Scientific Exchange Programme under JSPS-VCC programme (1988). He was a visiting scientist at Petronas Research and Scientific Services (1995). He has Research collaboration with Fritz Haber Institute, Max Planck Society, Berlin, Germany (2002). He was the Outstanding UKM Lecturer in Research and Teaching (2000, 2002 and 2005). He is a chairman of Xapp-MNS (X-ray Application Society), Malaysian Nuclear Society. He is a senior member of International Zeolite Association (IZA, USA), Malaysian Analytical Member Society (ANALIST) and Malaysian Nuclear Society.



Kamaruzzaman Bin Sopian obtained his BSc in mechanical engineering from the University of WisconsinMadison in 1985, MSc in energy resources from the University of Pittsburgh in 1989 and PhD. in mechanical engineering from the Dorgan Solar Laboratory, University of Miami in 1997. He is presently a professor in renewable energy at the Department of Mechanical and Material Engineering, Universiti Kebangsaan Malaysia.

Currently, he is the director of the Solar Energy Research Institute, a center of excellence for the research and development in solar energy technology. He has been involved in the field of solar energy for more than twenty years. His main contributions are in solar radiation modeling, alternative material for solar absorber, solar water heating system with integrated storage system, solar desalination, solar cooling, daylighting using solar light pipes, solar assisted drying systems, gridconnected photovoltaic system, thin film silicon solar cells, combined photovoltaic thermal or hybrid collector and solar hydrogen production system. He has published over 400 research papers in journals and conferences.