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Characterization of Adsorbent Developed from Rice Husk: Effect of Surface Functional Group on Phenol Adsorption

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Abstract: Over the last three decades, there has been increasing global concern over the public health impacts attributed to environmental pollution. Among various water pollutants, phenol and its derivatives are the most toxic because they are carcinogenic in nature. The objective of this study was to develop novel, low-cost adsorbent from rice husk modified by thermal and chemical treatments for the effective removal of phenol from industrial wastewater. The physical and surface properties of the developed adsorbents were characterized using FTIR, SEM and FSEM. The results indicated that the pretreatment of rice husk has caused the functional groups (-OH, Si-OH, C-H, C=O, C=C, CH₂, CH₃, CO, Si-O-Si, C-C, Si-H, -O-CH₃) on the surface of adsorbent to change and the specific surface area to increase. The adsorption capacity was greatly influenced by surface group changes. The effects of the presence of the surface functional group on adsorption of phenol were analyzed by observing the shifting of the FTIR peaks after the adsorption experiment. Analysis of FTIR shows that the -OH, C-H,-CO, C-OH, Si-OH and -Si-H groups contribute to the adsorption of phenol onto the surface of adsorbent.

Key words: Rice husk adsorbent, phenol adsorption, adsorbent functional group, treatment of wastewater

INTRODUCTION

Phenol has been known as a common and notorious contaminant in water environment. Phenol constitutes the 11th of the 126 chemicals, which has been designated as priority pollutants by the US Environmental Protection Agency (USEPA) (Nayak and Singh, 2007). The content of phenols in industrial wastewater in the range of 0.1-6800 mg L⁻¹ (Busca et al., 2008), is usually higher than the standard limit set by USEPA of 0.1 mg L⁻¹ and the standards set by Malaysia Environmental Quality Act, 1974 for Standard A (0.001 mg L^{-1}) (Ahmaruzzaman and Sharma, 2005). Human consumption of phenolcontaminated water can cause severe pain leading to damage of the capillaries and ultimately causing death. The most important sources of pollution are the wastewaters from the iron-steel, coke, petroleum, pesticide, paint, solvent, pharmaceutics, wood preserving chemicals, paper and pulp industries (Busca et al., 2008). Current methods for removing phenolic from wastewater include microbial degradation, adsorption on activated carbon, chemical oxidation, deep-well injection, incineration, solvent extraction and irradiation

(Aksu, 2005; Radetski et al., 2009; Jain et al., 2002). Among those methods, adsorption is a highly efficient technique which has gained considerable attention for treating aromatic compounds from aqueous streams. The efficiency of the adsorption process is mainly due to the characteristic of the adsorbent such as high surface area, high adsorption capacity, microporous structure and special surface reactivity. Despite of these advantages, adsorption process is expensive due to the high cost of the adsorbents and the regeneration process (Cherifi et al., 2009). Consequently, there has been a growing interest in developing and implementing various potential adsorbents for the removal of aromatic compounds from water and researchers are always in a hunt for developing more suitable, efficient, cheap and easily accessible types of adsorbents, particularly from the waste materials. Considerable efforts have been made by many researchers to search for cheaper substitutes such as bagasse pith, carbonized bark, peat, lignite, soil, chitin, rice husk, rice straw, wood and fly ash for the adsorption of phenols with varying success (Jain et al., 2002; Aksu, 2005; Ahmaruzzaman, 2008; Cherifi et al., 2009). The abundance and availability of agricultural

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by-products make them good sources of cheap raw materials for natural adsorbents. Rice husk, an agricultural waste, has been reported as a good adsorbent for many metals and basic dyes (Kumar and Bandyopadhyay, 2006; Wan Ngah and Hanafiah, 2008). Wan Ngah and Hanafiah (2008) reported that the pretreatments of rice husk increased the cellulose content of the solid fraction by virtue of lignin removal and hemicellulose solubilization. In addition pretreatment also caused the cellulose crystallinity to decrease and the porosity of the adsorbent to increase. Many researchers found that the modified rice husk exhibited higher adsorption capacities than unmodified rice husk (El-Shafey, 2007; Sahu et al., 2009). According to the statistics compiled by the Malaysian Ministry of Agriculture, there are more than 408,000 t of rice husk and rice straw produced in Malaysia annually (Chuah et al., 2005). Converting these materials into effective adsorbents can be an attractive option to eliminate waste materials. The present work focuses on the development of low-cost adsorbents from rice husk modified by thermal and chemical treatments for the effective removal of phenol from industrial wastewater. The effects of the presence of the surface functional group on the adsorption of phenol were also investigated.

MATERIALS AND METHODS

Preparation of adsorbent: The raw material was rice husk (RRH) obtained from a rice mill. The rice husk was washed thoroughly with distilled water to remove adhering soil and clay then dried in air at 105°C in an oven for 24 h. The rice husk was milled and then passed through different sieves. The particle size between 500-250 µm was selected for further pretreatment. Chemical treatments of RH were carried out using base, acid and organic compounds. Three different modification methods were selected: (1) base treatment using 0.5 M NaOH with stirring at room temperature (Kumar and Bandyopadhyay, 2006), (2) acid treatment using 13 M H₂SO₄ at 175-180°C for 20 min with stirring (El-Shafey, 2007) and (3) organic treatment by 1% formaldehyde in the ratio of (1:5 (rice husk:formaldehyde, w/v)) at room temperature for 24 h with stirring (Garg et al., 2009). The RH was washed with distilled water and dried at 50-60°C in electrical oven till the weight attained a steady state and constant value. Thermal treatments of RH were obtained by burning the rice husk at temperature of 300, 400 and 600°C to determine the optimum burning temperature. At 300°C considerable amount of carbon would be produced. However, at 400°C the amount of carbon decreased which cause an increase in the amount of silica and at 600°C high amorphous silica would be produced (Nair *et al.*, 2006). Four burning times of 1, 2, 3 and 4 h were selected to determine the optimum burning time. The adsorbents were characterized in term of the specific surface area using particle size analyzer (PSA, model MS2000), the morphological characteristics and chemical properties using scanning electron microscope (SEM, model LE01430VP) and field emission scanning electron (FSEM, model SUPRATM 55VP). In addition, the functional groups present in the adsorbents were determined using a Fourier Transform Infrared (FTIR, model 8400S) spectrophotometer. The spectra range chosen was from 4000 to 400 cm⁻¹.

Adsorption experiments: About 100 mg L⁻¹ of phenol solutions were prepared by dissolving an appropriate amount of phenol (purchased from Merck) in distilled water. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used to adjust the pH of the phenol solution. The adsorption experiments were carried out using the selected types of materials: (1) One gram of raw rice husk, (2) One gram of raw rice husk after pretreatment (3) 0.1 g of rice husk ash. The experiments were performed using 100 mL of phenol solution at pH 5.5. The suspensions in all sorption assays were stirred at room temperature for 24 h and then filtered to remove any suspended adsorbent. The functional group present in the materials was then analyzed.

RESULTS AND DISCUSSION

Effect of burning time: In order to examine the effect of heating time on the adsorbent produced, the heating time was varied from 1 to 4 h, keeping the heating temperature constant at 300, 400 and 600°C. In general, an increase in the burning time increased the amount of silica but decreased the amount of carbon content significantly. The decrease of carbon content will predictably cause the decrease of the porosity of the adsorbent. Figure 1 shows that as the burning time (at 300°C) increase from 1 to 4 h, the silica content also increase from 1.5 to 19.26 (wt. %). Consequently the carbon content has decreased from 73.77 to 41.21 (wt. %). This expected since at longer burning times, more carbon converted to silica. However, increasing the burning temperature to 400°C (Fig. 2) has shown different trend. The silica content was found to decrease for burning time from 1 to 2 h and consequently decreased the carbon content. However, further increase in the burning time from 3 to 4 h has caused the silica content to increase and the carbon content to decrease. This due to the reaction for the rice husk proceeded with the two stages. The first stage showed rapid mass decrease caused by cellulose decomposition at the first

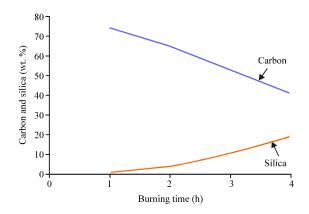


Fig. 1: Variation of weight percent of carbon and silica for RHA300 at different burning times

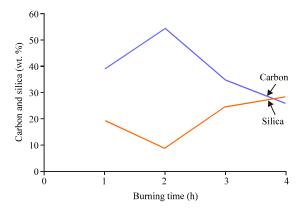


Fig. 2: Variation of weight percent of carbon and silica for RHA400 at different burning times

hour. Then, at the second stage, lignin decomposed for pyrolysis and its char burned for combustion (Gani and Naruse, 2007). The maximum decomposition rate of cellulose was at 350-400°C. Previous studies have shown that CO is formed during the primary decomposition of hemicelluloses and cellulose with a smaller portion of CO coming from lignin by the cracking of carbonyl (C-O-C) and carboxyl (C=O) in biomass (Wannapeera et al., 2008). The functional group analysis by FT-IR (the results are not shown) also confirmed that. Similar trend was observed for the burning temperature at 600°C (Fig. 3). However, the carbon content was not detected. Generally, the pretreatment of rice husk by chemical and thermal treatments reduced the content of hemicellulose, lignin and cellulose crystallinity which leads to an increase of the specific surface area for treated rice husk compared to raw rice husk (Table 1).

Characterization of adsorbent using FTIR: The chemical structure of the adsorbent is of vital importance in

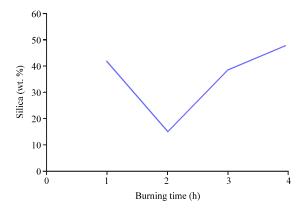


Fig. 3: Variation of weight percent of silica for RHA600 at different burning times

	Table 1:	Chemical	characterization	of	adsorb ent
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	Carbon	Silica	Oxygen	Specific surface
Adsorbent		(%)		area (m ² g ⁻¹)
Raw Rice Husk (RRH)	49.15	11.96	38.89	0.0127
RHA 300°C, 1 h	73.77	1.50	22.10	-
RHA 300°C, 2 h	64.47	3.82	30.83	-
RHA 300°C, 3 h	52.49	10.60	36.60	0.258
RHA 300°C, 4 h	41.21	19.26	39.53	0.415
RHA 400°C, 1 h	38.70	18.84	41.57	0.210
RHA 400°C, 2 h	53.67	8.64	34.20	0.415
RHA 400°C, 3 h	34.18	24.21	41.42	0.664
RHA 400°C, 4 h	25.14	27.89	43.92	0.717
RHA 600°C, 1 h	0.00	41.77	53.76	-
RHA 600°C, 2 h	0.00	15.10	54.21	-
RHA 600°C, 3 h	0.00	38.48	55.96	0.764
RHA 600°C, 4 h	0.00	48.15	47.87	0.845
RRH+13M H ₂ SO ₄	31.91	13.56	53.92	0.0226
RRH+0.5M NaOH	52.43	0.58	46.46	0.0137
RRH+Formaldehyde	46.22	4.73	49.05	0.0157

Table 2: Peak identification of FTIR spectra of raw and modified rice husk

Wave No. (cm ⁻¹)	Functional group
3404.31	-OH and Si-OH
2925.81	C-H stretching of alkanes
1641.31-1737.74	C=O stretching of aromatic groups
1546.8-1652.88	C=C stretching of alkenes and aromatic
1461.94	CH ₂ and CH ₃
1379.01	Aromatic CH and carboxyl-carbonate
1238.21	CHOH stretching of alcohol group
1153.35-1300	CO group in lactones
1080-1090	Si-O-Si
935.41	C-C
469-800	Si-H
580-34	-OCH ₃

understanding the adsorption process. The FTIR technique is an important tool to identify the characteristic functional groups, which are instrumental in adsorption of aromatic compounds. The FTIR spectra of the raw rice husk before and after phenol sorption were used to determine the vibrational frequency changes in the functional groups in the adsorbent (Table 2). As shown in Fig. 4, the adsorption peak around 3404.31 cm⁻¹ indicates the existence of free hydroxyl groups. In RRH,

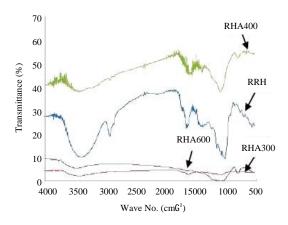


Fig. 4: FTIR spectra of RRH and RHA at different temperatures

the C-H stretching vibration around 2925.81 cm⁻¹ indicates the presence of alkane functional group. The peaks around 1641.31-1737.74 cm⁻¹ correspond to the C=O stretching that may be attributed to the hemicelluloses and lignin aromatic groups (Garg et al., 2009). The C=C stretching vibrations between 1546.80-1652.88 cm⁻¹ indicative of alkenes and aromatic functional groups. The peaks around 1461.94 cm⁻¹ indicate the presence of CH₂ and CH₃ groups while those at 1380 cm⁻¹ are indicative of CH₃ (Srivastava et al., 2006). A peak at 1379.01 cm⁻¹ band may be attributed to the aromatic CH and carboxyl-carbonate structures. The peaks in the 1153.35-1300 cm⁻¹ corresponds to vibration of CO group in lactones. The peaks around 1238.21, 1080.06 and 862.12-476.38 cm⁻¹, corresponds to CHOH stretching, Si-O-Si stretching and Si-H groups, respectively. The presence of polar groups on the surface is likely to provide the considerable cation exchange capacity to the adsorbent (Srivastava et al., 2006). As shown in Fig. 4, heating rice husk at 300, 400 and 600°C resulted in a loss of C-H stretching band (2925.81 cm⁻¹), C-C (935.41 cm⁻¹), C-O and C-O-C (1153.35, 1115 cm⁻¹) and C-O-H (899 cm⁻¹). These were replaced by the primary functional groups of C=O (1654.81, 1658.67, 1656.74, 1656.74 cm⁻¹) and silica functional groups of Si-O-Si (1099.35 cm⁻¹), Si-H (800.4 cm⁻¹) and Si-OH (3200-3700 cm⁻¹) (Thiravetyan et al., 2007). Figure 5-7, showed that the pretreatment of raw rice husk by 13 M H₂SO₄, 0.5 M NaOH and formaldehyde changed the functional groups onto the surface of adsorbent. Peaks in the region (3100-2800 cm⁻¹) are attributed to the stretching of carbon-hydrogen bonds. As shown in Fig. 8 and Table 3, the shifting of the peaks (from 3404.31, 2925.81, 1238.21, 1080.06, 709.76 and 574.75 cm⁻¹) were observed after phenol sorption. Based on these

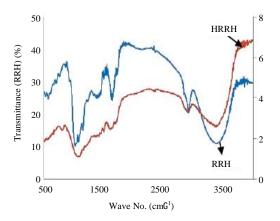


Fig. 5: FTIR spectra of RRH and RRH+13 M $\rm H_2SO_4$ (HRRH)

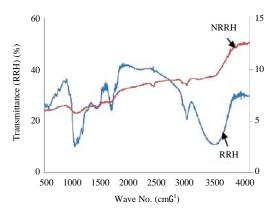


Fig. 6: FTIR spectra of RRH and RRH+0.5 M NaOH (NRRH)

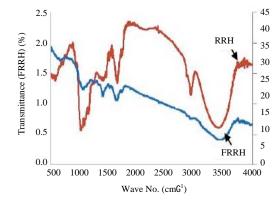


Fig. 7: FTIR spectra of RRH and RRH+Formaldehyde (FRRH)

observations, it can be stated that the functional groups (-OH, C-H, -CO, C-OH, -Si-OH and -Si-H) at these wave numbers have participated in the phenol adsorption.

Table 3: FT-IR spectra of RRH before and after phenol sorption Adsorbent Si-OH, -OH C=CC=0Si-O-Si Si-H RHA 3425.34 1600.81 1659.3 1099.35 678.90 RHA after 3382.91 1612.38 1655.3 1093.56 677.81 phenol sorption

Table 4: FT-IR spectra of RHA300 before and after phenol sorption						
Adsorbent	Si-OH, -OH	C=C	C=O	Si-O-Si	Si-H	
RHA	3421.48	1575.33	1714.69	1099.35	784.97	
RHA after	3423.41	1580.71	1712.67	1099.35	700.11	
phenol						
sorption						

Table 5: FT-IR spectra of RHA400 before and after phenol sorption					
Adsorbent	Si-OH, -OH	C-H	СНОН, -СО	Si-O-Si	Si-H
NRRH	3413.77	2921.96	1203.5	1041.49	896.8
NRRH	3404.13	2929.67	1209.2	1054.99	856.3
after phenol					
sorption					

Table 6: FT-IR spectra of RHA600 before and after phenol sorption					
Adsorbent	Si-OH, -OH	C-H	СНОН,-СО	Si-O-Si	Si-H
RRH	3404.3	2925.81	1238.21	1080.06	709.76
RRH after	3410.3	2927.74	1240.14	1081.99	707.83
phenol					
sorption					

Table 7: FT-IR spectra of RRH+0.5 M NaOH before and after phenol sorption

Adsorbent	Si-OH,-OH	C=O	Si-O-Si	Si-H
RHA	3423.41	1670.36	1093.56	802.33
RHA after				
phenol sorption	3417.63	1671.54	1089.71	800.40

Table 8: FT-I	R spectra of RI	RH+13M H	SO ₄ before and	after phenol	sorption
Adsorbent	Si-OH, -OH	С-Н	СНОН, -СО	Si-O-Si	Si-H
FRRH	3425.34	2858.31	1263.29	1056.92	827.4
FRRH	3419.56	2860.24	1267.14	1054.99	825.4
After phenol					
sorption					

Table 9: FT-IR spectra of RRH+Formaldhyde before and after phenol sorption

sor	ption				
Adsorbent	SiOH, -OH	C-H	C=O	Si-O-Si	Si-H
HRRH	3363.6	2920.03	1706.8	1099.35	563.18
HRH	3408.9	2929.67	1710.7	1101.28	561.25
after phenol					
sorption					

Figure 9-14 and Table 4-9, showed the fundamental peaks of all the adsorbents before and after phenol sorption for rice husk ash (300, 400, 600°C) and RRH modified by 0.5 M NaOH, 13 M $\rm H_2SO_4$ and formaldehyde, respectively. The rice husk modified by thermal and chemical treatments showed that shifting occurs at lower and higher wave numbers. This also confirms that the new functional groups on the surface of adsorbent affect the phenol sorption.

Characterization of adsorbents by using SEM and FSEM: The morphological characteristics of raw rice husk and modified rice husk was observed by using Scanning

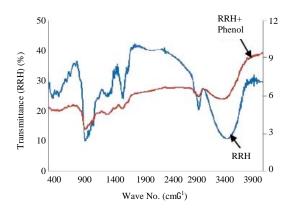


Fig. 8: FTIR spectra of RRH before and after phenol sorption

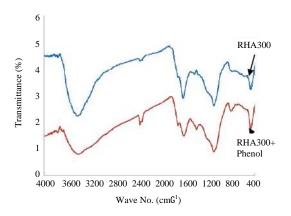


Fig. 9: FTIR spectra of RHA (300°C, 1 h) before and after phenol sorption

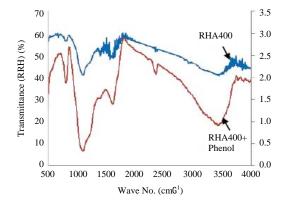


Fig. 10: FTIR spectra of RHA (400°C, 1 h) before and after phenol sorption

Electron Microscope (SEM) and field emission electron microscope (FSEM). Figure 16-18 show the successive micrograph of thermal treated rice husk produced at

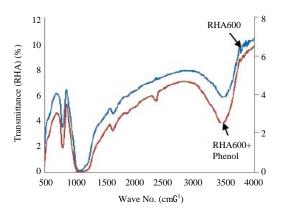


Fig. 11: FTIR spectra of RHA (600°C, 1 h) before and after phenol sorption

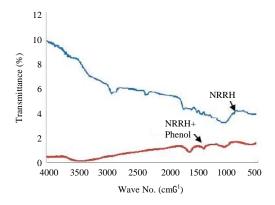


Fig. 12: FTIR spectra of RRH+0.5 M NaOH before and after phenol sorption

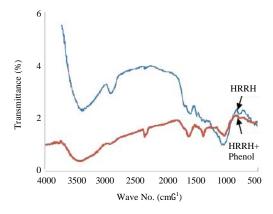


Fig. 13: FTIR spectra of RRH+13 M $\rm H_2SO_4$ before and after phenol sorption

different temperatures (300,400 and 600°C) at 3 h burning time. Figure 16 and 17 show the morphology of rice husk ash (300 and 400°C), respectively. The pores of different

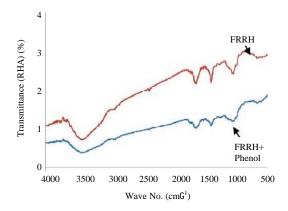


Fig. 14: FTIR spectra of RRH+ Formaldehyde before and after phenol sorption

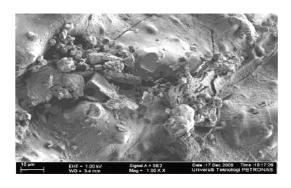


Fig. 15: FSEM for Raw Rice Husk (RRH), magnified 1000 times

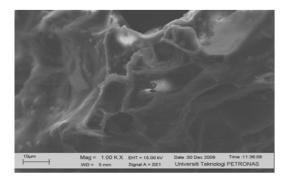


Fig. 16: SEM for rice husk ash (300°C, 3 h), magnified 1000 times

sizes and shapes could be observed compared to the raw rice husk (Fig. 15). According to the micrograph, the pores were developed from the decomposition of the RRH structure by heat and converted it to small particles with large surface area. The micrograph corresponding to

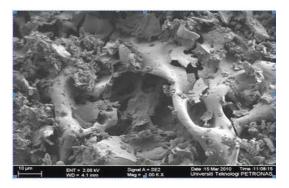


Fig. 17: FSEM for rice husk ash (400°C, 3 h), magnified 1000 times

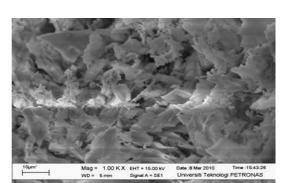


Fig. 18: SEM for rice husk ash (600°C, 3 h), magnified 1000 times

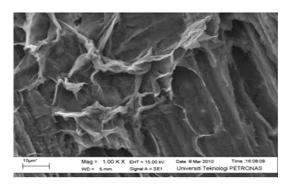


Fig. 19: SEM for raw rice husk +0.5 M NaOH, magnified 1000 times

calcination at high temperature (600°C) shows the loss of micropore volume, possibly attributed to pore collapse (Fig. 18). Figure 19 shows the morphological of treated rice husk with 0.5 M NaOH. After the treatment with the alkaline solution, the silica reacts with NaOH to form sodium silicate (Na₂SiO₃). The Na₂SiO₃ is soluble in water and is removed by adequate water-washing. As a result,

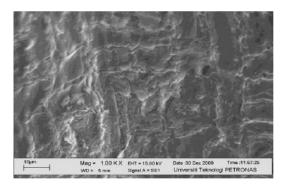


Fig. 20: SEM for raw rice husk +13 M H₂SO₄, magnified 1000 times

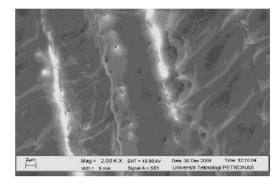


Fig. 21: SEM for raw rice husk+Formaldehyde, magnified 2000 times

some large holes remain on the husks outer epidermis (Liou and Wu, 2009). For rice husk treated by H₂SO₄ and formaldehyde (Fig. 20, 21), respectively, the porosity also increased compared to the raw rice husk, resulted from the removal of the inorganic compounds such as carbonate and silica from the surface of adsorbent. Consistent with the results reported by Lu and Lau (1996), samples activated with sulfuric acid generally have higher specific surface area and micropore areas than the non-activated adsorbent. Figure 19-21 also revealed that the activation of chemicals used significantly affect the surface area development and pore structure evolution.

CONCLUSIONS

The pretreatment of rice husk has caused the surface functional groups to change and the specific surface area of the adsorbent to increase. The surface functional groups have significant effect on the sorption of phenol on the surface of adsorbent. The RRH heated at 300°C contained considerable amount of carbon. However, the carbon content decreased with increased of burning

temperature. Consequently, this has led to an increase in the silica content. In addition, increasing the temperature from 300 to 600°C and burning time from 1 to 4 h, have resulted in a high porosity and large specific surface area of RHA compared to the RRH treated with base, acid and organic compounds. Analysis of FTIR shows that the -OH, C-H,-CO, C-OH, Si-OH and -Si-H groups contribute to the adsorption of phenol onto the surface of adsorbent. From both economics and environmental perspectives, these experimental results warrant further efforts, perhaps in terms of large scale manufacturing and testing.

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