



Research Article

Synthesis of KCC-1 Using Rice Husk Ash for Pb Removal from Aqueous Solution and Petrochemical Wastewater

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Abstract

A silica-rich rice husk ash (RHA, 95.44% SiO₂) was used as a silica precursor in the synthesis of KCC-1 for Pb(II) removal. The extraction of silica was carried out under several extraction methods (alkali fusion (AF), reflux (RF) and microwave heating (MW)) and extraction parameters (NaOH/RHA mass ratio, fusion temperature and H₂O/NaOH-fused RHA mass ratio). The highest silica content was obtained using AF method at extraction conditions of NaOH/RHA mass ratio = 2, fusion temperature = 550 °C, and H₂O/NaOH-fused RHA mass ratio = 4, with silica concentration of 85,490 ppm. TEM, FTIR, and BET analyses revealed the synthesized KCC-1 has fibrous morphology with surface area of 220 m²/g. The synthesized KCC-1 showed good performance in removal of Pb(II) from aqueous solution (74%) and petrochemical wastewater (70%). The analyses of petrochemical wastewater revealed that the adsorption process using synthesized KCC-1 effectively decreased the concentration of COD (489 mg/L to 106 mg/L), BOD (56 mg/L to 34 mg/L) and Pb(II) (22.8 mg/L to 6.71 mg/L). This study affirmed that KCC-1 was successfully synthesized using RHA as silica precursor and applied as an efficient adsorbent for Pb(II) removal. Copyright © 2019 BCREC Group. All rights reserved

Keywords: Rice husk ash (RHA); Alkali fusion; Lead; Adsorption

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1. Introduction

The issue of disposal of various pollutants into wastewater has been emerged with the rapid industrialization over these decades, with the heavy metals contributed to the largest sorts and even hardest to be treated. Lead (Pb(II)) undoubtedly as the most problematic element which brought adverse impacts to this ecosystem [1]. Even in a mild quantity, Pb(II)

can resulted in severe neurological and physiological consequences to human beings [1]. Once this element has been ingested exceeded the allowable concentration, it tends to accumulate in the human body, and consequently cause serious health disorders [2]. The widespread of Pb(II) in the ecosystem devoted to the efforts of investigating the most efficient technique to eliminate Pb(II) from contaminated water. There are several well-documented and widely applied heavy metals removal techniques such as precipitation stabilization [3], ion exchange [4], coagulation-flocculation [5], and adsorption [6–9]. Generally, the simplicity design, technical

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user friendly and cost effectiveness would be the key considerations for treatment selection [10].

In recent years, a considerable number of researches have been devoted the application of mesoporous silica materials as adsorbent due to their well-ordered structure and high surface area [11,12]. In 2010, a novel mesoporous silica, namely, fibrous silica nanosphere (KCC-1) with unique spherical shape and high surface area was discovered [13]. Unlike the typical pore-based silica materials, KCC-1 is surrounded by vast amount of dendrimer, thus form fibrous morphology on it [14]. The unique morphology of KCC-1 renders abundant accessible active sites, which subsequently enhance its performance towards several applications [15]. The impressive physical properties of KCC-1, induced the attempt to remove Pb(II) by using KCC-1 as adsorbent. Nonetheless, the involvement of relatively expensive commercial silica precursor during KCC-1 preparation such as tetraethyl orthosilicate ($\text{SiC}_8\text{H}_{20}\text{O}_4$, TEOS) and sodium silicate (Na_2SiO_3), gave the idea of seeking alternative silica source.

In the meantime, low cost and high availability agricultural by-products which owned high silica content would be a good choice. Rice husk, which known to be invaluable agro-based waste accounts for 545 million metric tons (roughly one-fifth of the annual yield of rice around the world) [16]. Factories tend to combust the rice husk due to the overwhelming availability of this rice husk to form rice husk ash (RHA) and dumped by landfilling. Since RHA was disposed in an uneconomical way, the utilization of RHA as silica precursor in KCC-1 preparation seem to be an attractive approach. Thus, in this study, RHA was used as silica precursor for KCC-1 and the performance of synthesized KCC-1 was analyzed for Pb(II) removal from aqueous solution and wastewater.

2. Materials and Method

2.1 Extraction of Sodium Silicate from Rice Husk Ash (RHA)

The rice husk ash (RHA) was collected from Kedah, Malaysia. Prior to the extraction process, acid leaching process was carried out by adding 10 g of RHA into 2 M HCl solution (100 mL), followed by stirring at 60 °C for 3 h. The mixture was filtered, and the collected filtrate was washed with distilled water, oven-dried (120 °C) and calcined (550 °C, 3 h). The obtained sample was named as acid-leached RHA (A-RHA). The composition of RHA and A-RHA was analysed using XRF.

The solution of sodium silicate from rice husk ash (Na_2SiO_3 -RHA) was prepared using A-RHA via several preparation methods (alkali fusion (AF) method, reflux (RF) method, and microwave heating (MW) method). For the AF method, the Na_2SiO_3 -RHA was prepared according to the method reported by Yilmaz and Piskin [17] with some modification. In brief, A-RHA (30 g) was mixed with sodium hydroxide (30 g, NaOH, Merck), followed by calcination (550 °C, 1 h) to form NaOH-fused RHA. The powder form of NaOH-fused RHA was mixed with deionized water and stirred for 24 h at room temperature. Then, the solution was filtered to obtain the filtrate (Na_2SiO_3 -RHA). For the RF method, the Na_2SiO_3 -RHA was prepared according to the method reported by Nayak and Bera [18] with some modification. In brief, A-RHA (30 g) was mixed with a solution of NaOH (30 g NaOH powder dissolving in 60 mL of deionized water), followed by heating for 3 h at 100 °C. The solution was stirred at room temperature for another 24 h, followed by filtration to obtain the filtrate (Na_2SiO_3 -RHA). Meanwhile, for the MW method, Na_2SiO_3 -RHA was prepared by referring to Kumchompoo *et al.* [19] with some modification. In brief, A-RHA (30 g) was mixed with a solution of NaOH (30 g NaOH powder dissolving in 60 mL of deionized water). The mixture was then being heated under microwave irradiation (600 W) at 100 °C for 5 min. The solution was stirred at room temperature for another 24 h, followed by filtration to obtain the filtrate (Na_2SiO_3 -RHA). All the Na_2SiO_3 -RHA were analysed using inductively coupled plasma mass spectrometry (ICP-MS) (Agilent 7500 Series), and the optimal amount of Na_2SiO_3 -RHA solution was used for the KCC-1 preparation.

2.2 Preparation of KCC-1

KCC-1 was prepared according to Le *et al.* [13] with some modification. In brief, Na_2SiO_3 -RHA, butanol (Merck, 1.5 mL), and toluene (Merck, 30 mL) were mixed in one beaker, whereas urea (Merck, 0.6 g), cetyltrimmonium bromide (CTAB, Aldrich, 1 g), and water (30 mL) were mixed in another beaker. Then, both solutions were mixed and stirred (30 °C, 45 min) before being heated in a Teflon-sealed hydrothermal reactor (120 °C, 5 h). The obtained solution was centrifuged, rinsed using distilled water, oven-dried (100 °C, 12 h), and calcined (550 °C, 5 h) to produce the KCC-1.

2.3 Characterization of KCC-1

The morphology of the synthesized KCC-1

was determined using Transmission Electron Microscopy (TEM Leo Libra-120). The functional groups were examined by Fourier-Transform Infrared (FTIR Spectrometer Nicolet iS5, Thermo Scientific) in the range of 4000 to 400 cm⁻¹. The textural properties of KCC-1 were analysis using Brunauer-Emmett-Teller (BET) (Micromeritics®) at 77 K.

2.4 Petrochemical Wastewater Analyses

Petrochemical wastewater was collected from Petrochemical Industry located in Pahang, Malaysia. The sample was analysed using ICP-MS (Agilent 7500 Series) to determine the concentration of heavy metals in the wastewater. Chemical Oxygen Demand (COD) analysis was carried out using DRB200 Reactor. Prior to the COD analysis, reagent was prepared using sulphuric acid (250 mL), distilled water (50 mL), potassium dichromate (2.4 g), mercuric sulphate (5 g) and argentum sulphate (4 g) [20]. The COD test was carried out by mixing the specific amount of reagent and wastewater, followed by preheated in DRB200 Reactor for 2 h at 150 °C. Then, the sample was analysed after cool down to room temperature. Biochemical Oxygen Demand (BOD) analysis was carried out using Dissolved Oxygen Meter (AZ-8403) with 5-day BOD standard method as reported by APHA_AWWA_WEF (2012).

Table 1. XRF Analysis of RHA and A-RHA

Parameter	RHA (%)	A-RHA (%)
SiO ₂	88.52	95.44
K ₂ O	5.783	2.43
P ₂ O ₅	2.76	1.02
CaO	1.35	0.54
MgO	0.46	0.18
Cl	0.40	0.18
SO ₃	0.24	0.00
Fe ₂ O ₃	0.16	0.10
MnO	0.14	0.08
Al ₂ O ₃	0.08	0.00
ZnO	0.04	0.01
Rb ₂ O	0.02	0.01
SrO	0.01	0.01
CuO	0.01	0.01
NiO	0.01	0.00

2.5 Adsorption Process

Batch adsorption experiments were executed on the Pb(II) removal from aqueous solution and petrochemical wastewater. For the case of Pb(II) aqueous solution, dissolution of an exact quantity of Pb(II) nitrate (Pb(NO₃)₂, Sigma-Aldich) in deionized water was carried out to prepare the stock solution. Thereafter, 1 g/L of KCC-1 was added to the Pb(II) solution (50 mg/L) under constant stirring at ambient temperature. Then, the samples were collected, centrifuged (3000 rpm, 2 min), and analysed using UV-vis spectroscopy at 520 nm. Similar procedure was carried out for petrochemical wastewater except for the preparation of stock solution.

The amount of Pb(II) adsorbed and Pb(II) removal percentage were computed using following equation:

$$q_t = \left(\frac{C_o - C_t}{m} \right) \times V \quad (1)$$

$$Removal (\%) = \left(\frac{C_o - C_t}{C_o} \right) \times 100 \quad (2)$$

where q_t (mg/g) stands for the value of Pb(II) adsorbed at particular time, C_o and C_t (mg/L) represent the initial concentration and the liquid-phase concentration of Pb(II) solution at particular time, respectively, V (L) stands for the volume of the Pb(II) solution and m (g) refers to the mass of KCC-1.

2.6 Regeneration and Reusability

The regeneration and reusability of the synthesized KCC-1 was studied using four consecutive adsorption-desorption cycles with 0.1 M HNO₃ as desorbing agent.

3. Result and Discussion

3.1 Pre-treatment of Rice Husk Ash

Table 1 shows the oxide groups in RHA with and without acid leaching treatment. From the result shown in Table 1, silica (SiO₂) is the major component in both RHA indicating the high suitability of RHA as silica precursor for KCC-1 synthesis. By comparing the percentage of SiO₂ in RHA and A-RHA, it was clearly observed that the pre-treatment of RHA with HCl was improved the purity of SiO₂ by removing the metallic impurities in RHA. The presence of chloride ion (Cl⁻) in HCl attracted the metallic element to form salts, which can be easily dissolved and removed by filtration [21].

The positive role of acid leaching in removing the metallic impurities of RHA was also reported by Bakar *et al.* with an increase in SiO₂ purity from 95.77% to 99.58% [22].

3.2 Sodium Silicate Preparation from Rice Husk Ash (Na₂SiO₃-RHA)

Figure 1 shows the influence of extraction method on the concentration of extracted silica. The amount of the silica content in Na₂SiO₃-RHA in a sequence of AF > RF > MW, indicating the excellent performance of AF method in the extraction process. An excellent performance of AF method was also reported by Shopert *et al.* [23], owing to its high silica concentration, efficient and energy-saving. Since AF method was found as a best method for extraction process, this method was used to identify the best extraction conditions.

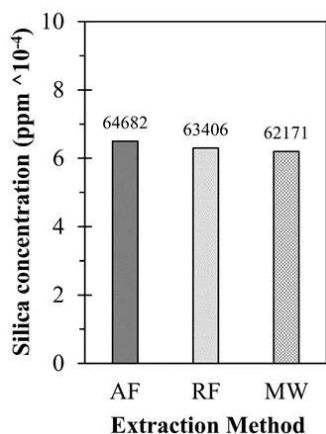


Figure 1. The silica concentration versus extraction method. Condition: NaOH/RHA = 1:1; T = 550 °C, and H₂O/NaOH-fused RHA = 2:1.

Several extraction parameters including NaOH/RHA mass ratio (5:1, 1:1, 2:1 and 3:1), fusion temperature (500, 550, 600 and 700 °C) and H₂O/NaOH-fused RHA mass ratio (1:1, 2:1, 4:1 and 5:1) were studied to identify the highest amount of silica concentration. Figure 2(A) shows the effect of NaOH/RHA mass ratio towards extracted silica concentration. An increase in the mass ratio of NaOH/POFA resulted to an increase in extracted silica concentration, passing through the optimum at 2, and decreased at elevated mass ratio NaOH/RHA. This result might be related with the changes in the pH of solution, and thus altering the silica dissolution process in aqueous solution which predominantly caused by the hydrolysis of Si–O–Si bonds [24]. Similar trend was reported by Keawthun *et al.* [25] for conversion of waste glasses into sodium silicate solutions.

Figure 2(B) shows the effect of the AF temperature towards extracted silica concentration. According to the plot, it was observed that increasing in AF temperature up to 550 °C, increased the concentration of extracted silica and slightly decreased at elevated temperature. The similar optimum fusion temperature (550 °C) was also reported by Yilmaz and Piskin [17] for the extraction of silica from tailings slurry of gold mine treatment plant by AF method. They reported that the most suitable fusion temperature is 550 °C and higher fusion temperature will lead to the degradation of chemical compounds and consequently decrease the silica extraction efficiency.

Figure 2(C) displays the effect of mass ratio of H₂O/NaOH-fused RHA towards the concentration of extracted silica. An increase in the mass ratio of H₂O/NaOH-fused RHA resulted

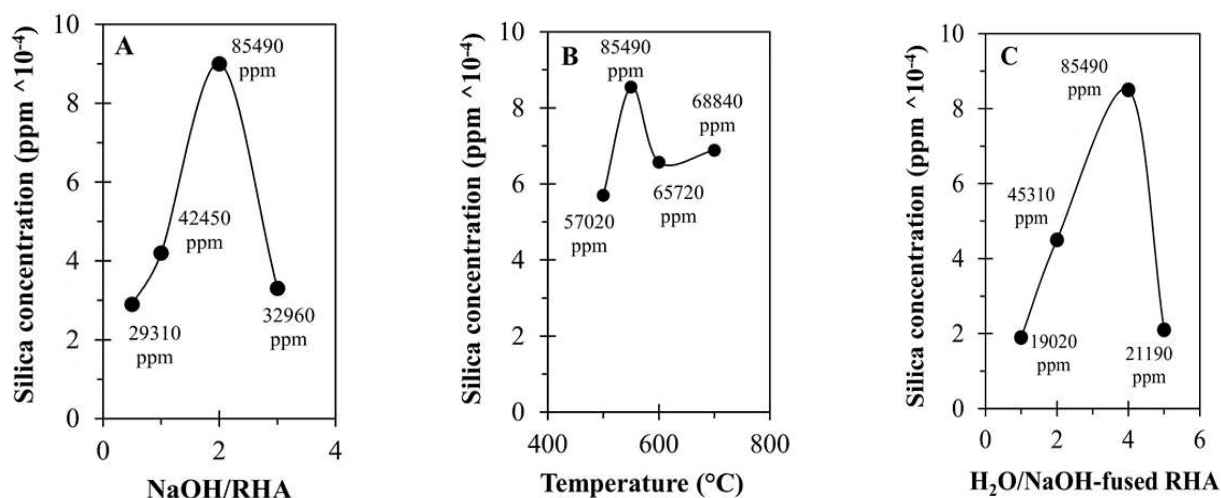


Figure 2. Concentration of extracted silica at different effect of (A) NaOH/RHA mass ratio; (B) fusion temperature; and (C) H₂O/NaOH-fused RHA mass ratio.

to an increase in extracted silica concentration, passing through the optimum at 4, and decreased at elevated mass ratio. Silva *et al.* [26] reported that the lower ratio of H₂O/NaOH-fused RHA would decrease the concentration of sodium, which can be claimed on the highly viscous solution worsen the leaching process. Similar trend was also reported by Shelke *et al.* [27] for the extraction of silica from RHA.

From the results observed in Figure 2, the best extraction conditions were achieved at mass ratio of 2, fusion temperature of 550 °C and H₂O/NaOH-fused RHA mass ratio of 4.

3.3 Characterization of KCC-1 Synthesized from RHA

Figure 3 shows the TEM image of KCC-1 synthesized from RHA. As illustrated from the figure, the sample was spherical in shape and covered with the fibrous morphology. The TEM image of the synthesized KCC-1 was in conformity with literature [15,28].

The N₂ physisorption analysis revealed that the synthesized KCC-1 possesses BET surface area (S_{BET}) of 220 m²/g, pore size (d_p) of 17.37 nm, and pore volume (V_p) of 0.94 cm³/g. As compared to the literature [29], the synthesized KCC-1 has relatively lower S_{BET} as compared to the literature (386 m²/g) that synthesized using TEOS, might be caused by the impurities that existing in RHA, thus, affected the KCC-1 structure [30]. This study was in agreement with Wang *et al.* [30] whom reported that the textural properties of SBA-15 synthesized using coal gangue (S_{BET} = 552 m²/g, d_p = 7.0 nm, and V_p = 0.54 cm³/g) was lower than that of

SBA-15 synthesized using commercial sodium silicate (S_{BET} = 567 m²/g, d_p = 7.2 nm, and V_p = 0.68 cm³/g), due to the impurities present in coal gangue.

The functional groups present in KCC-1 synthesized from RHA were analysed using FTIR, as shown in Figure 4. The spectra shows several bands at approximately 3423 cm⁻¹, 1058 cm⁻¹, 800 cm⁻¹, and 450 cm⁻¹, which ascribed to the O-H stretching vibration of Si-OH, Si-O asymmetrical stretching, Si-O symmetrical stretching, and bending of Si-O, respectively [31]. Similar peaks were also reported by Dong *et al.* (2015) for modified fibrous silica nanospheres [32], signifying the successful formation of KCC-1 structure from RHA as silica precursor.

3.4 Adsorption Performance of KCC-1 Synthesized from RHA

The adsorption performance of synthesized KCC-1 on Pb(II) removal was evaluated using aqueous solution and petrochemical wastewater and the results are shown in Figure 5. KCC-1 was proven to have high feasibility as adsorbent for Pb(II) removal as demonstrated in its high removal percentage (74% (aqueous), 70% (wastewater)), owing to its favourable structural properties. In addition, the analyses of wastewater (Table 2) clearly showed that the synthesized KCC-1 effectively reduced the COD, BOD, and heavy metals concentration of the wastewater, indicating an excellent performance of KCC-1 in adsorption process. An excellent performance of synthesized KCC-1 in Pb(II) removal (74%, 37.26 mg/g) can be proved

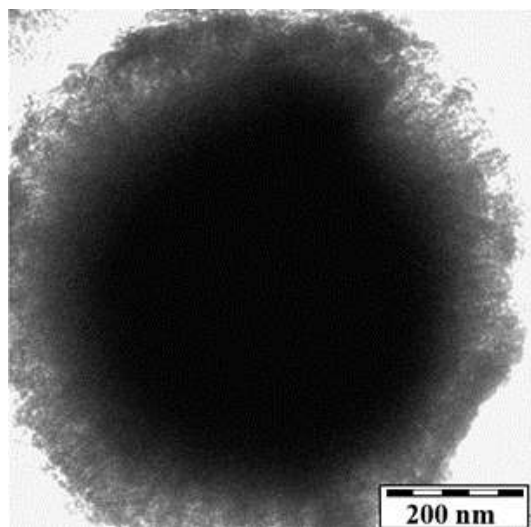


Figure 3. TEM image of KCC-1 synthesized from RHA.

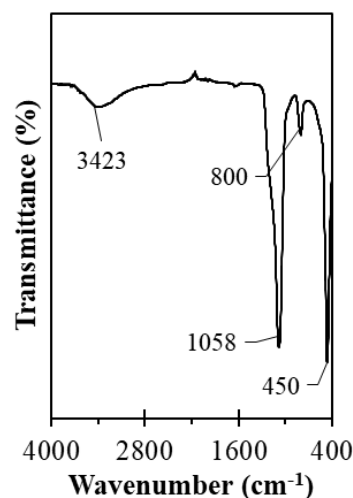


Figure 4. FTIR spectra of KCC-1 synthesized from RHA.

by the comparison of the KCC-1's performance with other reported adsorbent as listed in Table 3.

Figure 6 shows the regeneration and reusability of synthesized KCC-1 during four consecutive adsorption-desorption cycles. KCC-1 shows excellent performance during adsorption-desorption cycles with moderate decreased in the percentage of Pb(II) removal. The de-

clined in the adsorption performance with increasing in the number of cycles might be due to the blockage of some active sites available on the KCC-1's surface, owing to the partial desorption of Pb(II) molecules during the regeneration process [39]. The decreased in the adsorption performance of the synthesized KCC-1 (20 % for aqueous solution, 34 % for wastewater, 4 cycles) during consecutive adsorption-desorption cycles was lower as compared to the reported literatures for modified potato starch-magnetic nanoparticles (MPS-MNPs) (50%, 4 cycles) [40] and nanoscale zero-valent iron immobilized in alginate microcapsules (60%, 4 cycles) [41], implying good performance of synthesized KCC-1 in Pb removal.

The spent KCC-1 was analysed using FTIR spectroscopy and was compared with the fresh KCC-1 for identifying the functional groups

Table 2. Petrochemical wastewater analysis before and after the adsorption process using KCC-1 synthesized from RHA.

Parameter	Before (mg/L)	After (mg/L)
COD	489	106
BOD	56	34
Sodium (Na)	60.9	26.9
Potassium (K)	21.9	Not detected
Calcium (Ca)	4.2	3.34
Magnesium (Mg)	0.3	0.20
Copper (Cu)	0.0852	Not detected
Aluminium (Al)	0.0049	Not detected
Vanadium (V)	0.0031	Not detected
Manganese (Mn)	0.032	Not detected
Nickel (Ni)	0.0072	Not detected
Chromium (Cr)	< 0.0005	Not detected
Arsenic (As)	<0.0005	Not detected
Selenium (Se)	<0.0005	Not detected
Molybdenum (Mo)	0.936	Not detected
Silver (Ag)	<0.0005	Not detected
Cadmium (Cd)	0.0352	Not detected
Antimony (Sb)	0.0017	Not detected
Barium (Ba)	< 0.0005	Not detected
Lead (Pb)	22.8	6.71

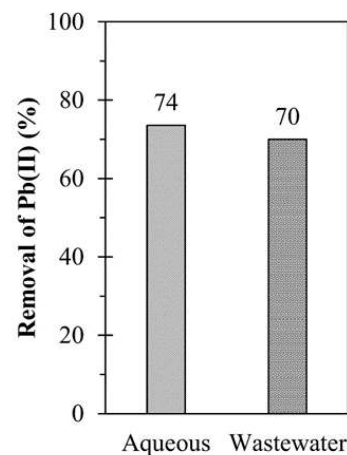


Figure 5. Removal percentage of Pb(II) from aqueous solution and petrochemical wastewater using KCC-1 synthesized from RHA. Conditions: $C_{0, \text{aqueous}} = 50 \text{ mg/L}$, $m_{\text{KCC-1}} = 1 \text{ g/L}$, $\text{pH} = 6$, and $\text{time} = 80 \text{ min}$.

Table 3. Comparison of adsorption capacity for KCC-1 with other reported adsorbents in Pb(II) removal.

Adsorbent	Adsorption capacity (mg/g)	Ref
KCC-1 synthesized from RHA	37.26	This study
MCM-41-S9	34.33	[33]
Diatomite	26.00	[34]
Silica Gel	14.42	[35]
Phenol-formaldehyde/silicon dioxide resin (PFSR)	13.74	[36]
Mercapto-modified silica particles	10.42	[37]
Anionic layered double hydroxide (LDH)	6.81	[38]

that responsible for the adsorption process (Figure 7). It was observed that the FTIR peaks of the synthesized KCC-1 (3423 cm^{-1} , 1058 cm^{-1} , 800 cm^{-1} , and 450 cm^{-1}) were shifted after the adsorption process due to the electrostatic and chemical interaction of functional groups with metal ions [42]. The significant alteration of 3423 cm^{-1} , in terms of the wavenumber and intensity might be related to the Pb molecules' interaction with Si–O–H of KCC-1 to form the Si–O–Pb.

4. Conclusion

The potential of KCC-1 synthesized from rice husk ash (RHA) was tested on Pb(II) removal from aqueous and petrochemical wastewater. Prior to the extraction process, acid treatment was executed to enhance the purity of silica, SiO_2 by elimination the metallic impurities in RHA. The study of extraction methods (alkali fusion (AF), reflux (RF) and microwave heating (MW)) and extraction parameters (NaOH/RHA mass ratio, fusion temperature and $\text{H}_2\text{O}/\text{NaOH}$ -fused RHA mass ratio) revealed that the highest silica content was obtained using AF method at extraction conditions of NaOH/RHA mass ratio = 2, fusion temperature = $550\text{ }^\circ\text{C}$, and $\text{H}_2\text{O}/\text{NaOH}$ -fused RHA mass ratio = 4, with silica concentration of 85,490 ppm. The characterization analyses (TEM, BET and FTIR) of synthesized KCC-1 in conformity with literature, signifying the successful formation of KCC-1 structure from RHA. The performance studies showed that the synthesized KCC-1 has a good performance in Pb(II) removal from aqueous (74%) and petrochemical wastewater (70%), with moderate reduction in the percentage of Pb(II) removal

during adsorption–desorption cycles. The wastewater analyses (COD, BOD and ICP-MS) revealed an excellent performance of KCC-1 as demonstrated by a significant decrease in the COD (489 mg/L to 106 mg/L), BOD (56 mg/L to 34 mg/L), and Pb(II) concentration (22.8 mg/L to 6.71 mg/L).

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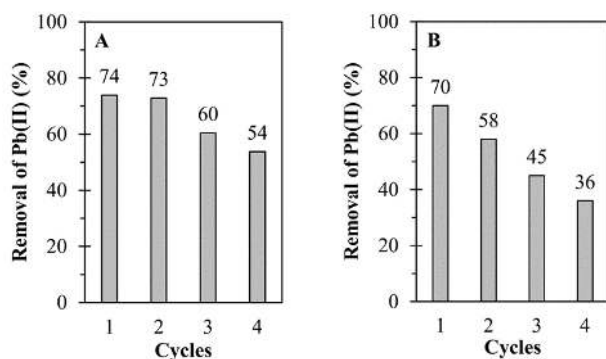


Figure 6. Reusability of KCC-1 synthesized from RHA during Pb(II) removal from (A) aqueous solution and (B) petrochemical wastewater. Conditions: $C_{0,\text{aqueous}} = 50\text{ mg/L}$, $m_{\text{KCC-1}} = 1\text{ g/L}$, and time = 80 min.

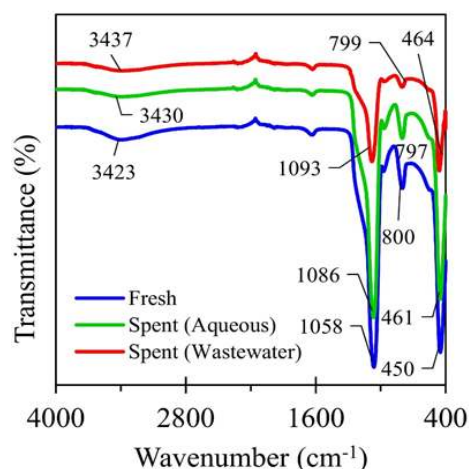


Figure 7. FTIR spectra of fresh and spent KCC-1 synthesized from RHA.

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