

A NOVEL RICE STRAW–BUTYL ACRYLATE GRAFT COPOLYMER: SYNTHESIS AND ADSORPTION STUDY FOR OIL SPILL CLEANUP FROM SEAWATER

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For the first time, a potential material for oil spill cleanup has been successfully synthesized by the graft polymerization of butyl acrylate (BA) onto rice straw (RS) using 2,2'-azobisisobutyronitrile (AIBN) as initiator and divinyl benzene (DVB) as cross-linking agent. The copolymer synthesis was controlled by the concentration of monomer BA and AIBN, reaction time, and temperature. It was found that the optimal conditions for the highest graft yield of 41.50% were as follows: [AIBN] = 0.04 mol/L, [BA] = 1.50 mol/L, 180 min at 75 °C. The graft copolymer RS-g-BA was structurally analyzed by Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), X-ray diffraction (XRD), and thermogravimetric analysis (TGA). In the presence of DVB (1.5%), the maximal adsorption capacity of the copolymer was 20.56 g/g. The reusability of the RS-g-BA copolymer was assessed during seven sorption/desorption cycles with 70% recovery. The experimental data were clearly more suitably fitted by the Langmuir monolayer adsorption model rather than by the Freundlich isotherm model. The kinetic study indicated that oil adsorption by RS-g-BA is likely to be a chemisorption process.

Keywords: graft copolymer, rice straw, butyl acrylate, oil spill cleanup

INTRODUCTION

Oil is one of the most important energy sources, which is used as a raw material for obtaining fuel and various products. There has been an increasing demand for oil supply in the modern industrial world. However, oil spills may occur during the production, transportation, storage and use of this resource, causing major environmental problems that directly affect aquatic life, local economy, tourism, and leisure activities. Thus, oil sorbents, which can be produced from synthetic and natural products, have become a necessity. At present, the most common materials used for oil sorbents are synthetic ones, but they are considered non-biodegradable and are relatively expensive. Thus, the search for oil sorbents developed from natural resources has drawn much scientific interest.

Rice straw (RS) is an agricultural residue available in large quantities, which, at present, has limited uses. Currently, a vast amount of RS is not exploited as an industrial raw material, most of it being discarded as waste or burned. This not only leads to the loss of a potentially valuable raw material, but also causes environmental harm. Hence, the utilization of renewable agricultural residues, such as RS, has become the focus of scientific concern.^{1,2} Many attempts to create eco-friendly and biodegradable products using RS as a raw material have been proposed. For example, in a study by Y. Huang *et al.*, alkali treated RS reinforced geopolymer composites have been developed.³ Other researchers carried out the modification of RS by microwave heating and established that the obtained adsorbent material

might remove 90% of Cd^{2+} and Cr^{3+} metal ions in 60 min, and 99% of Pb^{2+} in 30 min.⁴ In another study, RS was converted into an aerogel, which reached an oil adsorption capacity of up to 13 g/g.⁵

Graft polymerization is a synthesis process during which side chains are covalently linked to a polymeric backbone, aiming to enhance the advantageous physical or chemical properties of polymers. Most of the lignocellulosic graft polymerizations are followed by a radical reactive mechanism, in which vinyl derivatives and AIBN are the most commonly used monomeric agents and initiators, respectively.⁶ Among vinyl monomers, the hydrophobic monomer BA is suitable for the synthesis natural-based copolymers for application as adsorbents, as it promotes the swelling behavior and sorption efficiency of products.⁷ It is also evident that the inclusion of cross-linkers, such as DVB derivatives, into the lignocellulosic graft copolymers directly affects their oil sorption capacity.⁸

Many attempts have been made to develop oil spill sorbents derived from natural-based materials due to their low cost, abundance, and environmental friendliness. Nevertheless, their main disadvantages, such as low oleophilic/hydrophobic features, represent a significant obstacle.⁹ Therefore, chemical modification of these raw materials is necessary. The current research aims to synthesize a novel oil sorbent via grafting polymerization of monomeric BA onto the cellulosic backbone of RS. DVB and AIBN have been employed as cross-linker and initiator, respectively (Fig. 1). The FTIR, SEM, XRD, and thermal analyses were used to analyze the graft copolymer RS-g-BA. Langmuir and Freundlich isotherms were

examined to describe the adsorption process, whereas the pseudo-second-order model was found to fit most suitably the adsorption data.

EXPERIMENTAL

Materials

Rice straw (RS) was collected from Donganh, Hanoi, Vietnam, in May 2020. The RS sample was dried in an oven at 60 °C for 24 h. It was then cut into small pieces of 1-2 mm, and extracted with methanol-benzene (1:1, v/v) for 6 h to remove wax and resin. BA, AIBN, DVB, and absolute ethanol were of analytical laboratory grade. Crude oil was supplied by the Research Center for Petroleum Exploration and Production – Vietnam Petroleum Institute.

Synthesis

The method used for synthesis has been carefully described in previous publications.¹⁰⁻¹² The reaction was carried out under nitrogen atmosphere in a 250 mL three-neck flask, equipped with a stirrer and a condenser. RS (1.0 g) was immersed in 15 mL of DMF for 4 h, and pre-calculated amounts of monomer BA and cross-linker DVB were added to the flask. The system was purged with nitrogen gas during 10 min to remove oxygen, and then heated under stirring. A pre-calculated amount of initiator AIBN was slowly added to the reaction mixture and stirred well. After 3 h, the mixture was poured into 100 mL of absolute ethanol to precipitate the product, and then washed several times with absolute ethanol to remove residual monomers. Finally, the graft copolymer RS-g-BA was dried under vacuum at 60 °C to constant weight. The percentage of graft yield (GY) was determined by the following formula:

$$\text{GY (\%)} = \frac{W_2 - W_1}{W_1} \quad (1)$$

where w_2 and w_1 are the weights of RS-g-BA and RS, respectively.

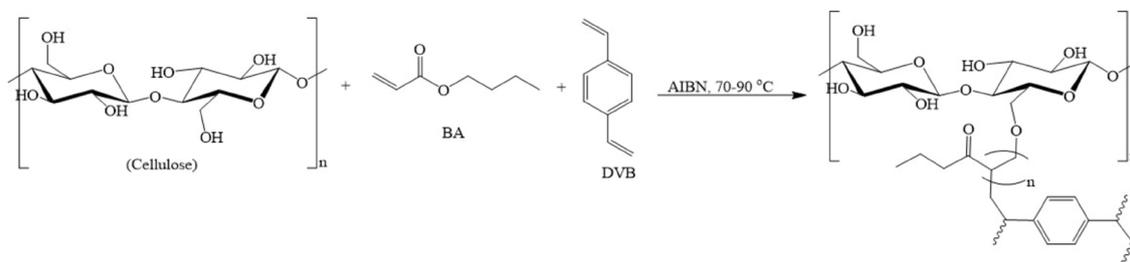


Figure 1: Plausible grafting reaction of RS-g-BA

Characterisation

A Nicolet FTIR spectrometer (IMPACT 410, USA) was used to record the IR spectra in the range of 4000-400 cm^{-1} . The samples were mixed with dried KBr powder. A scanning electron microscope (JEOL 6390, Japan) was used to study the morphological features of

the material. A Siemens Bruker D5000 X-ray powder diffraction system (D5000, USA) was used for the X-ray diffraction studies. The samples were scanned between 5° and 50°. Thermogravimetric analysis was performed on a Labsys TG-DSC 1600 thermal analyzer (Setaram). All samples were analyzed in air

atmosphere, at a heating rate of 10 °/min from room temperature to 800 °C.

Oil absorption

Crude oil was introduced into a beaker containing seawater.^{13,14} The sorbent RS-g-BA (2.0 g) was placed into the oil layer and gradually stirred at 105 rpm. After a certain period, the sorbent was removed and drained for 2 min before weighing. The following equation was used to calculate the oil sorption capacity (Q):

$$Q \text{ (g/g)} = \frac{(C_0 - C_t) \times V}{m} \quad (2)$$

where C_0 and C_t stand for the concentrations of oil sorption (g/L) at $t = 0$ and t , respectively.

Reusability

The reusability of the adsorbent was evaluated following the method described in a previous report.¹¹ After adsorption, the sorbent was removed from the liquid using a mesh screen and vacuumed. Both the sorbent and the recovered oil were weighed after vacuum suction. In total, seven sorption/desorption cycles were performed.

RESULTS AND DISCUSSION

Effect of reaction time

The effect of reaction time on the GY has been outlined in Figure 2. In general, the GY increases linearly with reaction time, and reaches a maximum at 180 min. The GY rate is not constant beyond 180 min. Understandably, the increase of the GY in the early stage of the reaction is caused by the high concentration of BA and a large number of grafting sites.¹⁴ During a longer reaction time, homopolymeric reactions among monomeric BAs may take precedence over copolymeric reactions.¹⁴ As a result, a reaction

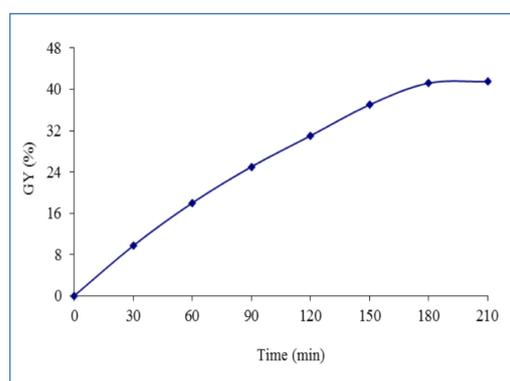


Figure 2: Effect of reaction time on GY; [BA] = 1.50 mol/L, AIBN = 0.04 mol/L, reaction temperature = 75 °C

time of 180 min can be regarded as optimum for the maximal GY.

Effect of temperature

The effect of reaction temperature on the copolymer grafting process of monomeric BA onto cellulosic RS is described in Figure 3. As reaction temperature increases from 70 to 90 °C, the GY first increased, reaching a maximal amount of 41.50% at 75 °C. However, the GY was found to decrease beyond 75 °C. The diffusion of monomer and initiator towards the cellulosic backbone and their interactions with cellulose macroradicals accelerated, as the reaction temperature rose in the first stage (70-75 °C).⁷ However, the GY decreased as the reaction temperature rose further (75-90 °C), which was most likely caused by the formation of an unstable complex formed by the initiator or to the dominance of the termination reactions.⁷

Effect of monomer concentration

As shown in Figure 4, the monomer concentration was varied from 0.5 to 2.0 mol/L, causing the GY to fluctuate accordingly. The highest GY of 41.50% was linked to the BA concentration of 1.50 mol/L. When the BA concentration was beyond this amount, the GY was observed to go down.

According to Khullar *et al.* (2008), increased availability of grafting sites to monomer BA leads to an increase in the GY in the early stages of the reaction.¹⁵ However, the reduced rise of the GY beyond the optimal BA concentration could be related to the competition between grafting copolymeric and homopolymeric reactions in the final stage of the reaction.¹⁵

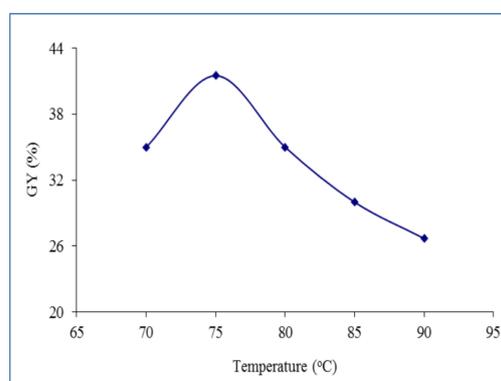


Figure 3: Effect of temperature on GY; [AIBN] = 0.04 mol/L, [BA] = 1.50 mol/L, reaction time = 180 min

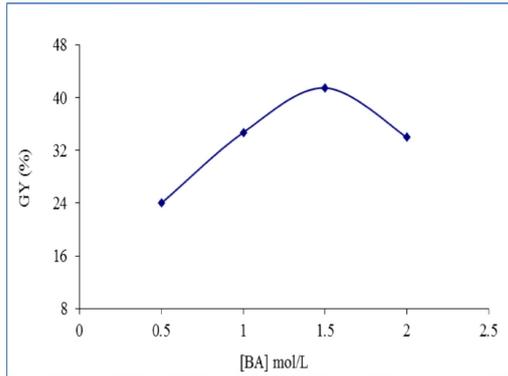


Figure 4: Effect of monomer concentration on GY; [AIBN] = 0.04 mol/L, reaction time = 180 min, reaction temperature = 75 °C

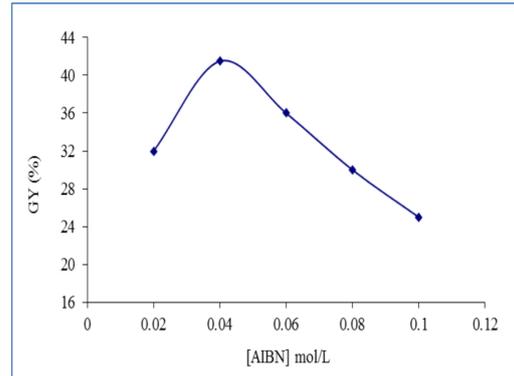


Figure 5: Effect of initiator concentration on GY; [BA] = 1.50 mol/L, reaction time = 180 min, reaction temperature = 75 °C

Effect of initiator concentration

The effect of the initiator AIBN concentration on the GY was observed in the range from 0.02 to 0.10 mol/L (Fig. 5). Similarly to the trends described above, the GY increased and accounted for the highest percentage of 41.50% when the AIBN concentration increased from 0.02 to 0.04 mol/L. At a higher initiator concentration of above 0.04 mol/L, the GY gradually decreased. AIBN in the range of 0.02-0.04 mol/L has played a crucial role promoting radical formations of methylene groups in poly(BA) and hydroxyl groups in the cellulose macromolecule.^{14,16}

Nevertheless, the concentration of initiator AIBN beyond 0.04 mol/L is responsible for a reduction in the grafting process, since homopolymeric reactions of BA facilitated the viscosity of reaction system.^{14,16}

FTIR spectroscopy

As shown in Figure 6 (a and b), the IR spectrum of the copolymer RS-g-BA exhibits significant differences compared to that of the raw material.

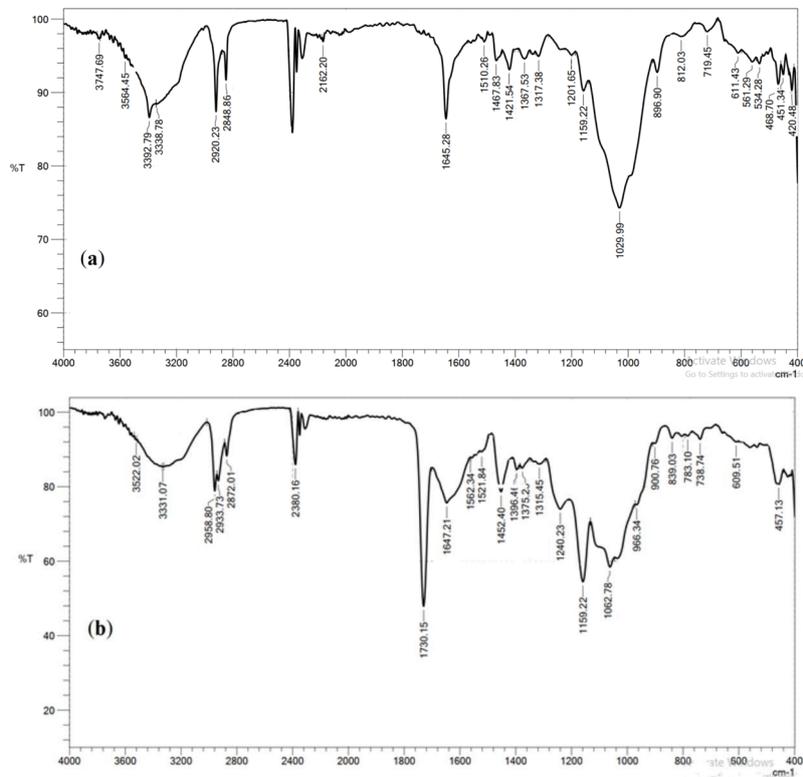


Figure 6: FTIR spectra of RS (a) and RS-g-BA (b)

An intense broad peak absorption around 3330.0 cm^{-1} , due to OH stretching vibrations of cellulose, and the peaks around 2900.0 cm^{-1} that can be assigned to C-H stretching vibrations of methyl and methylene groups are observed in the spectra of both materials.^{17,18} Also, the broad peaks at 1029.99 and 1062.78 cm^{-1} in the respective IR spectra of RS and RS-g-BA stand for C-O valence vibrations.¹⁸ Of particular importance is a new sharp peak around 1730.15 cm^{-1} appearing in IR spectrum of RS-g-BA, which is due to carbonyl stretching of an ester, which provides strong evidence of a successful grafting process.

Scanning electron microscopy (SEM)

As can be observed in SEM micrographs (Fig. 7), the untreated RS surface is coarse, because of

the presence on its surface of naturally occurring plant wax. After graft polymerization, the BA was homogeneously distributed on the RS surface and the material became a homogeneously porous polymer network. This may allow the material to retain different solvents in the matrix.

X-ray diffraction (XRD)

As may be seen in Figure 8, the XRD pattern of RS exhibits two peaks at 22.7° and 18.0° with the relative intensities of 135.1 and 90.2 (a.u.), respectively. These two peaks have disappeared in the pattern of copolymer RS-g-BA, and only a broad peak at 22.1° with the relative intensity of 120 (a.u.) can be observed. It can be concluded that the inclusion of poly(BA) side chains to the backbone of the polymer leads to a decrease in crystallinity.¹⁹

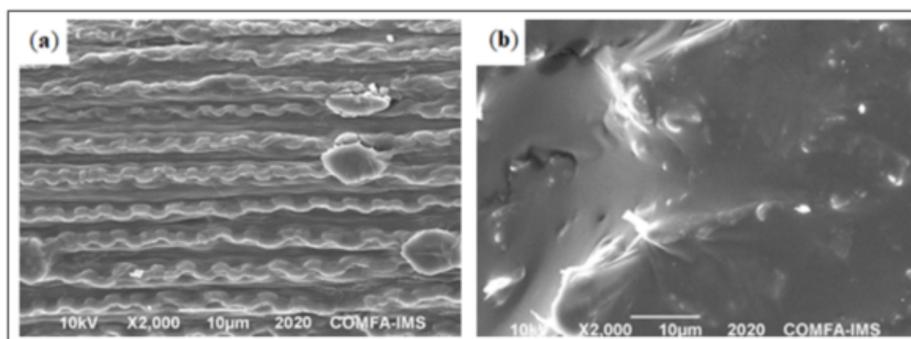


Figure 7: SEM images of RS (a) and RS-g-BA (b)

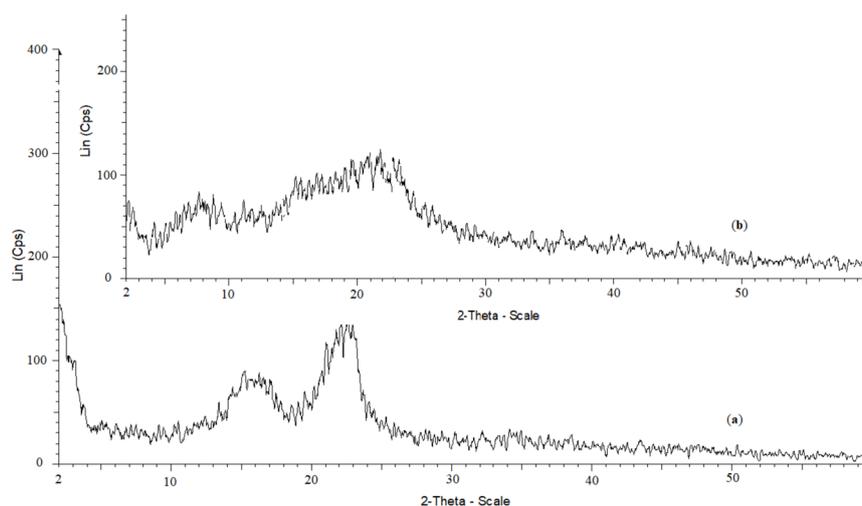


Figure 8: XRD patterns of RS (a) and RS-g-BA (b)

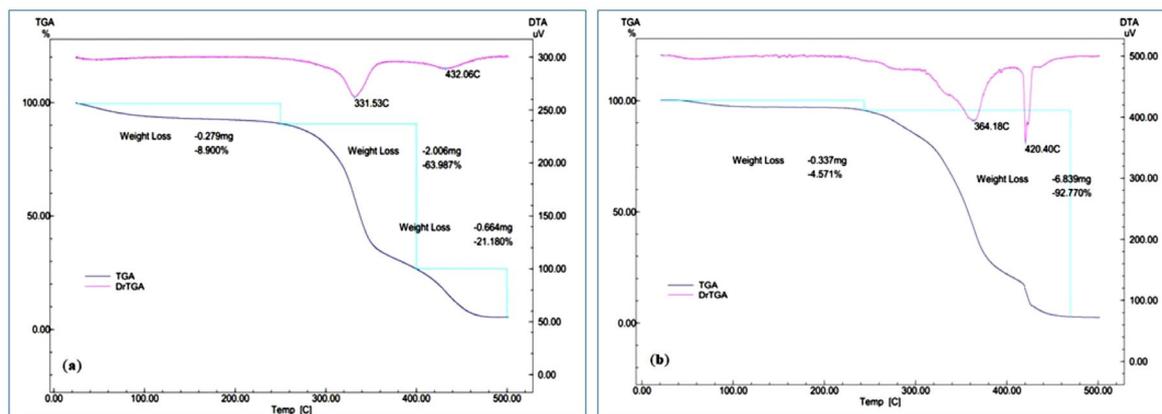


Figure 9: TGA curves of RS (a) and RS-g-BA (b)

Thermal analysis

The decomposition of RS and RS-g-BA is contingent upon changes of temperature. As can be seen from Figure 9, the weight loss of RS is due to water liberation in the first stage, up to 250 °C. However, the decomposition of cellulose fibrils is observed in the range from 250 to 395 °C, especially at 331.53 °C. RS shows solid residues at 500 °C.²⁰ When considering RS-g-BA, the first degradation stage of this copolymer is noted below 230 °C (Fig. 9). This suggests that cellulosic copolymer materials can fully meet the requirements set for a variety of applications.²⁰ The weight loss assigned to the decomposition of cellulose fibrils happened beyond 230 °C, particularly at 364.18 °C. Significantly, comparing the TGA curves of the materials, that corresponding to the copolymer RS-g-BA exhibited an additional peak at 420.40 °C. It represents the weight loss due to the degradation of the grafted poly(BA). This finding confirms the successful grafting formation.

Effect of cross-linking agent and contact time on oil sorption capacity and reusability

After discovering the optimum conditions for obtaining the maximum grafting yield of RS-g-BA, we moved on to finding out the best DVB content for reaching the best sorption capacity (Q) of the material. Actually, the cross-linking agent used is one of the key factors in the successful formation of a 3D structural network of polymers. In this context, DVB may be thought as the best cross-linking molecule to improve the sorption capacity and swelling behavior of oil sorption materials.^{21,22}

In this study, the DVB content was varied from 0 to 2.50% (Figs. 10-11). According to the

results, Q reached 15.0 g/g without any DVB, while the maximal value of 20.56 g/g was associated with the DVB content of 1.50%. However, a decrease in Q was recorded when the DVB content rose beyond 1.50%. An excess of cross-linking agent will result in the production of a copolymer network that is overly dense, and will substantially reduce the chain length between cross-linking locations, reducing the mobility of the polymeric chains.²¹ This is unfavorable for oil penetration inside the network of the sorbent, thereby the oil sorption is low. As compared with previous reports, our copolymer recorded a Q value much higher than other similar products. Indeed, the crude oil sorbent based on grafting of BA onto coir fiber was associated with the maximal Q value of 13.45 (g/g),²³ or the copolymer obtained from the combination of BA and banana fiber exhibited the maximal Q value of 14.45 (g/g).¹⁰

The dependence of Q on contact time (5-35 min) is illustrated in Figure 12. The Q of the RS material did not show any changes with the increase of time. In contrast, the crude oil sorption of RS-g-BA increased drastically, and reached the highest rate of 20.56 g/g at 25.0 min. It turned out that beyond 25.0 min, the Q value presented no changes. This effect might be explained by rapid sorption on the surface of the adsorbent in the initial stage of the process, followed by a slower adsorption stage occurring by penetration into the inner microscopic voids of the adsorbent.²⁴

According to the data listed in Table 1, the Q values of RS and RS-g-BA decreased with the number of reuse cycles. Because of the poor elasticity and irreversible deformation of the RS, its Q value for the 7th cycle was only 34.78% of that recorded in the 1st cycle. As regards the RS-

g-BA copolymer, after being reused for seven cycles, it maintained about 70% of its original oil absorption capacity. This phenomenon can be explained by the fact that monomers help maintaining the elasticity and capillarity of the adsorbent. The mechanical properties of RS-g-BA were not much affected after vacuum suction; thereby its reusability was much improved, compared to that of RS.

Equilibrium adsorption studies

Adsorption isotherm studies

Many approaches to studying the adsorption phenomenon are known, but the Langmuir and Freundlich isotherm models are often considered

as appropriate for describing single-component systems.^{10,25-27} The Langmuir isotherm assumes that the adsorption occurs at specifically homogeneous sites within the adsorbent.^{10,25-27}

The linear Langmuir equation is the following:

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{C_e}{q_{\max}} \quad (3)$$

where C_e represents the oil equilibrium concentration (g/L) in solution, q_e is the oil amount adsorbed at equilibrium (g/g), q_{\max} is the monolayer capacity of the adsorbent (g/g). The Langmuir constant K_L demonstrates the affinity between the adsorbent and the adsorbate.

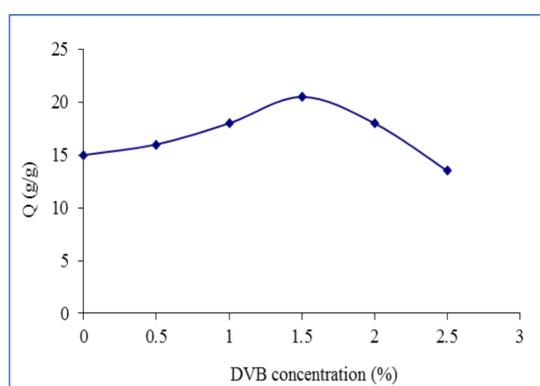


Figure 10: Effect of cross-linking agent content on oil adsorption

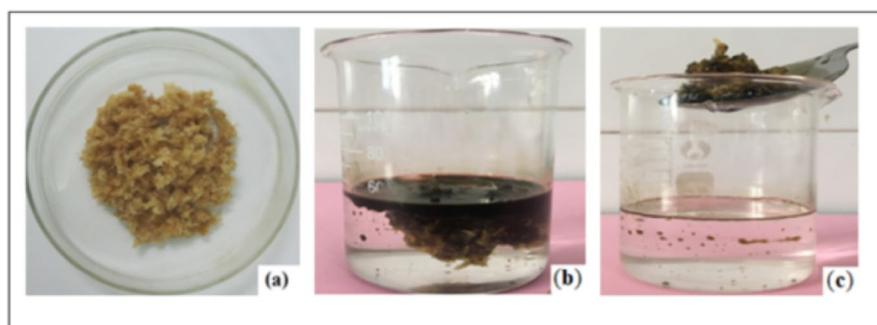


Figure 11: Pictures for cleanup of crude oil from water; (a) Copolymer RS-g-BA, (b) oil adsorption onto RS-g-BA, (c) removal of oil-loaded RS-g-BA

Table 1
Reusability of RS and RS-g-BA

Cycle	Crude oil sorption	
	RS	RS-g-BA
1	1.61	20.56
2	1.02	19.21
3	0.85	18.56
4	0.82	17.42
5	0.71	16.24
6	0.62	15.03
7	0.56	14.42

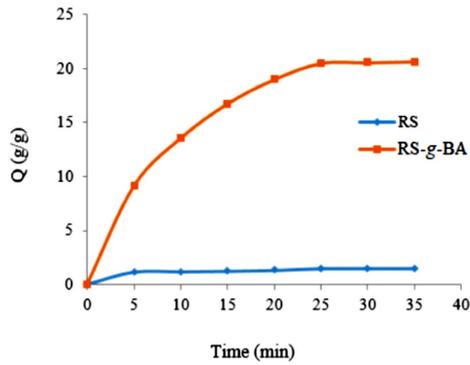


Figure 12: Effect of sorption time on oil sorption capacity of RS and RS-g-BA

The Freundlich isotherm equation is an empirical model used to describe heterogeneous systems that are not limited to monolayer formation:

$$\text{Log } q_e = \frac{1}{n} \text{Log } C_e + \text{Log } K_F \quad (4)$$

where K_F and n are Freundlich constants, showing the adsorption capacity and adsorption intensity of the sorbent, respectively.

The adsorption isotherm of RS and RS-g-BA was established at 25 °C. The adsorption capacity of these two materials was found dependent on C_o (Fig. 13). As the oil concentration increased, the adsorption capacity of RS-g-BA also increased and reached a plateau after $q_e = 20.56 \text{ g/g}$. From Figure 13, it can be further concluded that the adsorption of RS-g-BA is much better than that of the raw RS material.

To facilitate the prediction for the adsorption capacity, experimental data were fitted to the Langmuir and Freundlich isotherm models. The Langmuir model is characterized by the C_e/q_e versus C_e plot, whereas $\log C_e$ against $\log q_e$ is plotted for the Freundlich model (Fig. 14). It

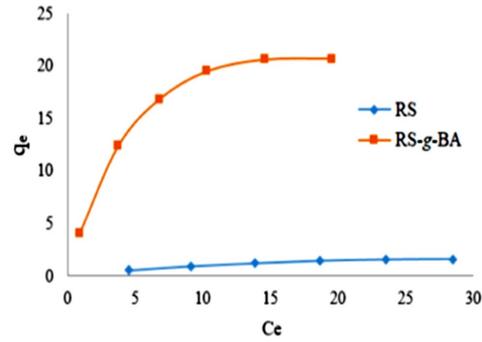


Figure 13: Adsorption isotherm of RS and RS-g-BA (C_e – oil equilibrium concentration (g/L), q_e – adsorbed oil amount at equilibrium (g/g))

should be noted that the adsorption is significant as the slope n ranges from 2.0 to 10.0.²⁷ The closer the coefficient R^2 is to 1.0, the more suitable is the model for describing the adsorption data.²⁷ According to the data tabulated in Table 1, the adsorption of RS and RS-g-BA are more suitably described by the Langmuir model, theoretically indicating monolayer adsorption, rather than by the Freundlich model.

Adsorption kinetics

To investigate the adsorption kinetics of RS and RS-g-BA materials, the pseudo-second-order model appears to be the best approach.²⁵⁻²⁷ The linear equation of this model is:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \quad (5)$$

where K_2 is the second-order rate constant (g/g.min).

A plot of t/q_t versus t will be linear if the adsorption is governed by the pseudo-second-order model.²⁵⁻²⁷ The slope and intercept can be used to compute q_e and K_2 , respectively.

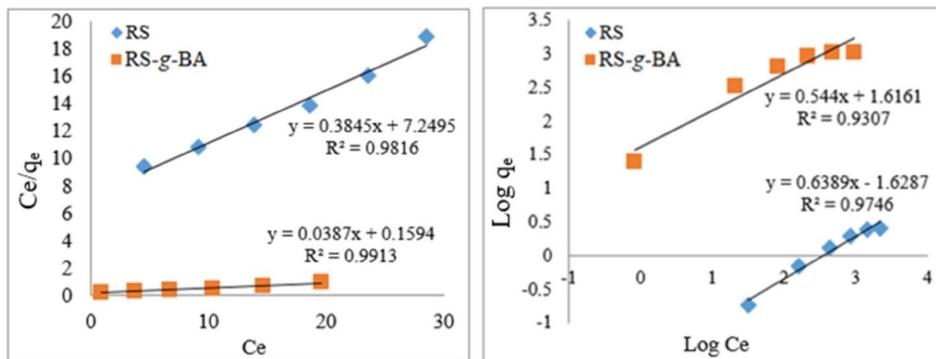


Figure 14: Langmuir and Freundlich isotherm models for adsorption of crude oil onto RS and RS-g-BA

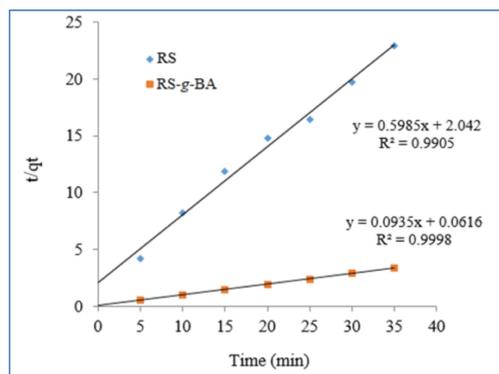


Figure 15: Pseudo-second-order equation model for adsorption of crude oil onto RS and RS-g-BA (q_t – adsorbed oil amount at time t (g/g))

Table 2
Adsorption data according to Langmuir, Freundlich and pseudo-second-order models

Sample	Langmuir model			Freundlich model			Pseudo-second-order model		
	K_L	q_{max}	R^2	K_F	n	R^2	K_2	q_e	R^2
RS	0.706	1.61	0.9816	7.730	9.276	0.9635	0.0587	3.4395	0.9571
RS-g-BA	2.278	20.56	0.9913	6.233	4.3253	0.969	0.0361	8.4890	0.9795

Figure 15 presents the pseudo-second-order plot for the adsorption of crude oil at various contact times. According to the results listed in Table 2, the rate constant K_2 scaled down from 0.0587 to 0.0361 g/g.min, when RS was converted into RS-g-BA. It means that the adsorption process happens inside the pores of the adsorbent matrix and is quick and reliable.²⁷ A linear relationship of t/q_t and t is observed for both RS and RS-g-BA. With the correlation coefficient R^2 of 0.9795, the sorption process of oil onto the RS-g-BA copolymer can be considered chemisorption.²⁷ Thus, the butylated cellulosic material should be suitable for oil sorption.

CONCLUSION

For the first time, the RS-g-BA oil sorbent was synthesized by graft polymerization of BA onto RS, using initiator AIBN and cross-linking agent DVB. The optimal conditions for obtaining the highest GY of 41.50% were the following: [BA] = 1.50 mol/L, [AIBN] = 0.04 mol/L, 180 min, at 75 °C. The successful formation of the copolymer was investigated by FTIR, SEM, XRD and thermal analyses. The maximal sorption capacity of the RS-g-BA copolymer towards crude oil reached 20.56 g/g, with the presence of 1.50% DBV content and for a contact time of 25.0 min. With 70% recovery, the synthesized RS-g-BA copolymer was reused during with seven sorption/desorption cycles. Equilibrium

adsorption studies indicated that the sorption process was best described as monolayer adsorption and chemisorption.

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