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Preparation of Banana Fiber Dye Adsorbent Through Radiation Induced Grafting

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Abstract : The present work reports preparation of natural fiber, banana fiber, dye adsorbent by using pre-irradiation grafting technique. Banana fibers were chemically pre-treated with sodium chlorite solution to remove the lignin and used the fiber as trunk polymer. Monomer, Glycidyl methacrylate (GMA), was grafted on pre-treated banana fibers by electron beam pre-irradiation technique and then followed by functionalization with imidazole to prepare the polymeric dye adsorbent. Dye adsorption test was carried out by using Acidic Blue 80 dye as modal dye. Grafting and functionalization were characterized by FTIR and SEM. Preparation of banana fiber adsorbent through radiation induced grafting was effectively carried out in this work.

Index Terms **- radiation-induced grafting, electron beam accelerator, banana fiber, Glycidyl methacrylate (GMA), dye adsorbent.**

I. INTRODUCTION

Dyes are widely used as colouring agent in many industries. Since the most dyes are complex, the discharge of dye effluents from industries into the water bodies creates potential threat to the aquatic lives. Thus, removal of dye from wastewater is crucial before it is released into water bodies. Several technologies have been used for dye removal including chemical method, adsorption, membrane separation and coagulation (Siti Kartina Abdul Karim et al, 2016). Adsorption is a physical process and has the advantages over the other methods because of simple design and low cost. Many research works have been reported various adsorbent materials holding a variety of attached chemical functional groups. Among them, the radiation method is a simple, accurate and relatively cleaner process to produce grafted polymeric adsorbent because of additives or catalysts are seldom required for the initiation, easy to control, homogeneous,.etc. (Sarala Selambakkannu et al, 2019). Radiation induced grafting can be performed by mutual irradiation and pre-irradiation technique (C.V.Chaudhari et al, 2016). This work was carried out by using pre-irradiation technique.

Banana fiber was used as truck polymer in this work. Recently, different agricultural wastes have been used to develop the adsorbent materials. Banana fibers are widely available around the world as a kind of agricultural waste from banana cultivation and so adsorbent materials development from this fiber will be beneficial.

This work focused on preparation of dye adsorbent through pre-irradiation induced grafting of GMA on banana trunk fibers followed by amine group functionalization. The adsorption test of dye was carried out in batch mode by using acidic blue 80 dye as a modal dye..

II. MATERIALS AND METHOD

2.1 Materials

The lignocellulosic banana fiber is used in this work and analytical grade chemicals of $NaClO_2$ (Sigma Aldrich, Malaysia), Nitric acid (Sigma Aldrich, Malaysia), glycidyl metharylate (GMA) (Sigma Aldrich, Malaysia), surfactant polyoxyethylene sorbitan monolaurate (Tween 20) (Sigma Aldrich, Malaysia), Imidazole (Sigma Aldrich, Malaysia), Acidic Blue 80 were provided by Radiation Processing Technology Division, Malaysian Nuclear Agency. All chemicals were used as received.

2.2 Pre-treatment of Banana Fiber

Before grafting, the banana fiber was pre-treated for the partial removal of lignin. 0.8% concentration of sodium chlorite ($NaClO_2$) solution was prepared in distilled water. The pH of $NaClO_2$ solution was adjusted to pH4 by adding nitric acid in a

fume chamber. Prepared $NaClO_2$ solution was heated up to 70°C on a hot plate stirrer. Banana fiber was added to the solution and remained in it for 6 hours. Thereafter, the banana fiber was removed from the solution and washed repeatedly with distilled water and dry in oven at 60°C until to get stable weight.

2.3 Preparation of Monomer Emulsion

GMA, a water insoluble monomer, was dispersed in water using surfactant, Tween-20, to form stable emulsion. The monomer emulsion was prepared with GMA 3% and Tween-20 1% in distilled water. Before grafting, the emulsion was purged with nitrogen for an hour to remove the dissolved oxygen.

2.4 Grafting of GMA onto Pre-treated Banana Fiber

GMA was graft polymerized on pretreated banana fiber by pre-irradiation technique using electron beam accelerator. The known weight of dry pretreated banana fiber was placed in polyethylene plastic zipper bag. The air inside the plastic bag was removed by purging with N_2 and sealed the bag as quickly as possible. The samples were preserved under dry ice temperature and irradiated with electron beam (EB) accelerator (NHV-EPS 3000) at different pre- determined doses at the energy of 2 MeV and 10 mA of current. The irradiated fiber and then 100 ml of monomer emulsion were transferred into glass vacuumed ampoules under vacuum condition. The glass ampoules were left in temperature controlled water bath for grafting reaction. The grafting reaction time was 3 hrs at 40°C in water bath. Upon completion of the reaction, samples were removed from emulsion and washed with methanol thoroughly to remove excess monomer and homopolymer. Thereafter, samples were dried in an oven at 50°C for overnight. After getting the constant weight, the weight of GMA-grafted banana fibers were measured. The degree of grafting, grafting yield (%) was calculated by using the following formula:

$$\text{Grafting yield (\%)} = [(W_g - W_i)/W_i] \times 100\% \quad (1)$$

Whereby, W_i and W_g are the weight of banana fiber before and after grafting process (C.V.Chaudhari et al, 2016).

2.5 Functinalization of GMA-grafted-banana Fiber

For the functionalization process, imidazole solution was prepared with 30% imidazole in 70% methanol. 0.5g of GMA-grafted-banana fiber which has attained maximum grafting yield was placed in an ampoule and immersed in a 60ml of imidazole solution. The ampoule was then placed in a water bath for 2 hrs at 70°C for the chemical reaction to take place. Subsequently, the functionalized banana fibers were washed with distilled water thoroughly. The washed fibers were placed in an oven to dry at 50°C. Thereafter, the density of functionalized groups was determined by the following equation:

$$\text{Amine [mmol/g-adsorbent (ad)]} = (Z_f - Z_i)/Z_i/M \times 1000 \quad (2)$$

Where by Z_i and Z_f are the weight of the fiber before and after functionalization and M is the molecular weight of imidazole (Sekine, A. et al, 2010).

2.6 Characterization of Fiber

Pristine, pretreated, GMA-grafted and amine functionalized fibers were analyzed by Fourier Transformed Infrared spectroscopy (Platinum ATR-FTIR, Tensor II, Bruker). FTIR spectra were measured in the range from 400 cm^{-1} to 4000 cm^{-1}, with a resolution 4 cm^{-1} and an average of 16 scans. The morphology analysis of pristine, pre-treated, grafted and functionalized fibers was done using FEI Quanta 400 scanning electron microscope (SEM). Sputtered gold coating was done on fibers via Bio Rad system to obtain good quality SEM images.

2.7 Preparation of Dye Solution

In the present work, dye adsorption test was carried out using Acidic Blue 80 dye. A stock solution of 1000mg/L of Acidic Blue dye solution was prepared by dissolving required amount of dye into the distilled water. The required concentration of dye solution was prepared by diluting the stock solution with distilled water.

2.8 Dye Adsorption by Functionalized GMA-grafted-fibers

Dye adsorption with different initial concentration was carried out in batch mode. The predetermined initial concentrations were 10, 20, 30, 40 and 50ppm. 0.05g of functionalized GMA- grafted- fiber adsorbents were tested by immersing into 50ml of dye solution in plastic bottles. Plastic bottles with adsorbent and dye solution were simultaneously and continuously stirred on a magnetic stirrer for 3hrs. After adsorption test, the absorbance of these solutions before and after 3hrs was determined using UV/visible spectrophoto-meter (DR-5000) at λ_{max} of 625nm. The dye concentration was calculated from a calibration curve. The removal of dye was calculated by using the following equation:

$$\text{Removal (\%)} = (C_o - C_e/C_o) \times 100 \quad (3)$$

Whereby, C_o and C_e are the initial and final concentration of Acidic Blue 80 dyes in the solution (S. Suganyaa et al, 2017)

III. Results And Discussion

3.1 Effect of Radiation Dose on Grafting Yield

The effect of irradiation dose on grafting yield was studied at 3 different doses of 10, 50 and 100 kGy. As shown in Fig.3.1, the grafting yield (GY) sharply increases for the former points and decline later point. At 10 kGy, GY was 68% and increased to 81% at 50kGy. At 100kGy, GY declined to 78%. When banana fibers are exposed to e-beam, high energy electrons are bombarded to the cellulose backbone of fibers. The active radicals will be formed by transferring the energy to OH molecules of the cellulose backbone. These active radicals are very short lived species and can only preserved at temperature of -22 °C. In the presence of GMA monomer, the micro radicals were added onto fibers to the double bond of GMA. This will lead to the formation of covalent bond between monomer and fiber which in turn, will propagate grafting onto irradiated fibers. By increasing the irradiation dose, more electrons are bombarded with cellulose and more radicals are formed and so grafting yield is increased. In this work, GY at 100 kGy was slightly decreased because of homopolymer formation. During experiment, some homopolymer were found visually on banana fibers. The higher concentration of radicals increases their recombination and leading to homopolymerization (Yongxia Sun and Andrzej G. Chmielewski, 2017).

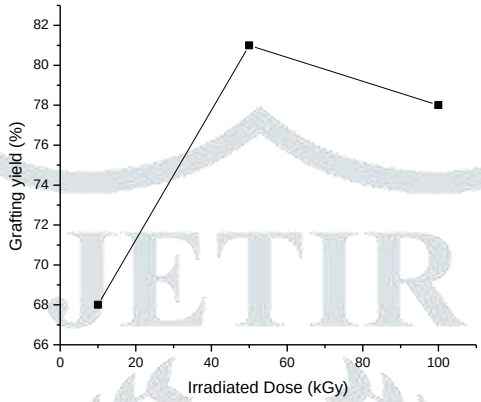

Figure 3.1: Effect of Irradiation Dose on Grafting Yield (%) of Banana fiber

3.2 Functionalization Results

The GMA-grafted-banana fiber with maximum GY of 81% was functionalized with imidazole. During functionalization reaction, the amine groups of imidazole attached to epoxy groups on the grafted GMA chains. The amine density was obtained 2.2787 mmol/g-ad for this work

3.3 FTIR analysis

FTIR analysis was conducted on prinstine, pre-treated, GMA-grafted and imidazole functionalized banana fibers. Figure 3.2 display FTIR spectra for prinstine and pre-treated banana fiber showing the peaks of interest for this work. Both prinstine and pre-treated fibers show the peaks responsible; for cellulose, hemicellulose and lignin at 3341 cm^{-1} (O-H stretching absorption) and at 2900 cm^{-1} (C-H stretching), for hemicellulose and lignin at 1720 cm^{-1} (C=O stretching), for lignin at 1635 cm^{-1} (C=C stretching and for cellulose at 1030 cm^{-1} (C-O stretching) (S. Suganyaa et al, 2017). The formation of major peaks does not change but the peak intensity reducesfor pre-treatment fiber. It's the evidence of removal of lignin, hemicellulose and cellulose with $NaClO_2$ treatment.

Figure 3.3 exhibits FTIR spectra for pre-treated and GMA-grafted (GY-81%) banana fiber. GMA grafting occurance was confirmed by an elongated 1722 cm^{-1} peak which was assigned to C=O vibration indication the existence of ester group –COO- contribution from GMA monomer. Emergence of epoxy characteristics peak at 1253, 904 and 842 cm^{-1} also proved successul grafting of GMA onto banana fiber (Sarala Selambakkannu et al, 2014).

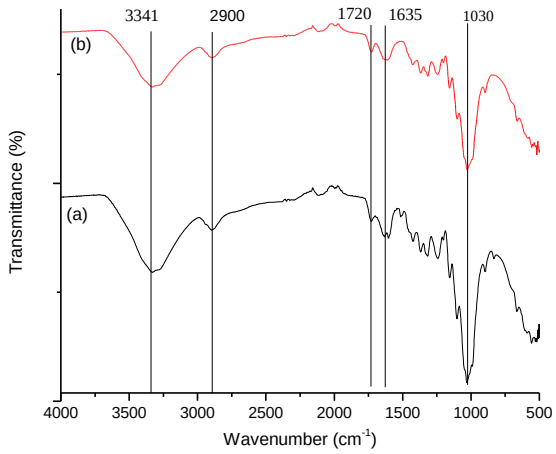

Figure 3.2: FTIR Spectra of (a) Prinstine Banana Fiber and (b) Pre-treated Banana Fiber

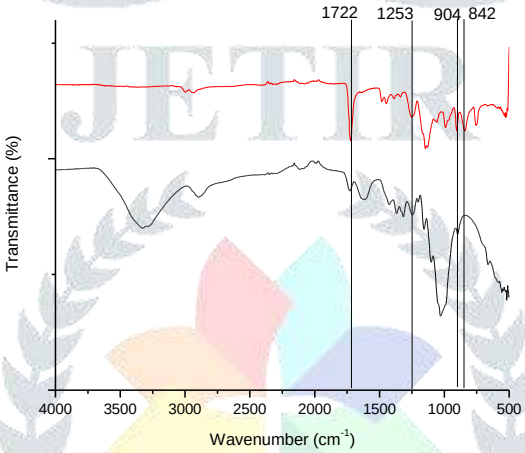

Figure 3.3: FTIR Spectra of (a) Pre-treated Fiber and (b) GMA-grafted Fiber (GY-81%)

Figure 3.4 illustrates the FTIR spectra for GMA-grafted (GY-81%) and imidazole functionalized-banana fiber. The peak of 1722 cm^{-1} assigned to C=O was observed on both spectra but the reduction of peak intensity was found for functionalized fiber. The vanishing of epoxy characteristics peak at 1253, 904 and 842 cm^{-1} for functionalized fiber supports the epoxy site of GMA were used by imidazole functional groups. Appearance of N-H stretching at 3340 cm^{-1} and N-H bending at 1567 cm^{-1} on functionalized fibers were confirmation of amine functioinalization (Selambakkannu, S. et al, 2015)

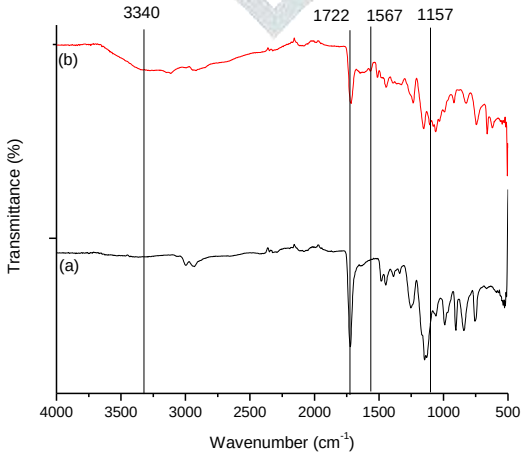

Figure 3.4: FTIR Spectra of (a) GMA-grafted Fiber, (GY-81%) and (b) Imidazole-functionalized Fiber

3.4 SEM Analysis

Prinstien banana fibers were chemically treated with $NaClO_2$ to remove the lignin which is highly deposited between microfibrils. Figure 3.5 illustrates the SEM images of (i) prinstine, (ii) pre-treated fibers, (iii) GMA-grafted and (iv) imidazole-

functionalized fibers respectively. It is clearly seen that the binding materials cover the prinstine banana fiber thoroughly, as a result the individaul fibers are held together as a bundle form (Khalil, H.P.S. et al, 2010). It can be clearly seen at (ii), during chemical treatment, the deposited lignin was soluble and clearly defined micro-fibers were appeared (Saha, S.C. et al). The formation of thin layer on banana fiber shown in Fig. 3.5 (iii) was the evidence of GMA graft co-polymerizat-ion onto banana fibers. After functionalization with imidazole, the fiber was coated with think layer as illustrated at Fig.3.5 (iv).

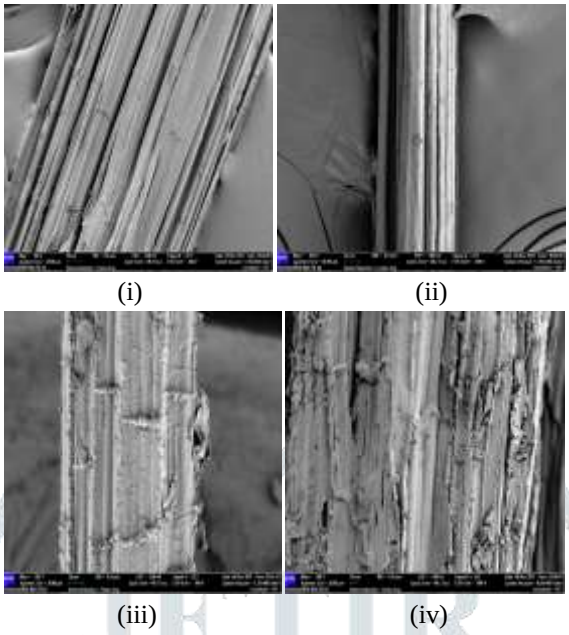

Figure 3.5: SEM Images of Banana Fibers,
(i) Prinstine Fiber, (ii) Pre-treated Fiber
(iii) GMA- grafted Fiber (GY-81%), (iv) Imidazole-functionalized Fiber

3.5 Dye Adsorption Results

Development of dye adsorbent was confirmed by dye adsorption test. The removal of dye from dye solution for different initial concentrations was shown in Table 1. It can be seen that for 10ppm concentration of dye solution, the removal % was 62% and at higher dye concentration, the adsorbent efficiency was low and it require more mass of adsorbent to increase the removal percentage.

Table 3.1. Removal (%) of Dye for Different Initial Concentration of Acidic Blue 80 Dye Solution

Dye Content	Removal of Dye (%)
10 ppm	62
20 ppm	47
30 ppm	36
40 ppm	32
50 ppm	30

IV. Conclusion

In this work, the dye adsorbent was successfully prepared form banana trunk fiber through radiation-induced grafting followed by functionalization. The GMA-grafted-fiber and imidazole functionalized fibers were verified by characterization with FTIR analysis and SEM spectroscopy imaging. The functionalized fibers were proven its ability in removal acidic Blue 80 dye. It can be conclude that radiation induced grafting banana fiber has the potential to be used as adsorbent for certain dyes.

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