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PREPARATION AND CHARACTERIZATIONS OF MAGNETIC RUBBER WOOD (*Hevea Brasiliensis*)

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ABSTRACT

Magnetic wood was prepared from rubber wood using *in situ* precipitation method. Magnetite (Fe_3O_4) nanoparticles were synthesized using Fe^{2+} and Fe^{3+} and NaOH into the rubber wood structure. Magnetic rubber wood prepared via *in-situ* method possessed ~20% of loading degree as compared to the subsequent loading method which only possessed ~9%, after an impregnation time of 24 hours. The electron micrographs showed the presence of magnetic particles deposited into the wood structure and atomic absorption spectrometer (AAS) was utilized to observe the particle distribution of magnetite deposited into the wood structure. Thermo gravimetric analysis (TGA) was used to evaluate the thermal decomposition and the activation energy (Ea) of the magnetic rubber wood block.

INTRODUCTION

The properties and utilization of rubber wood have been studied since the 1950's; however the commercial utilizations were only begun in 1970's. Afterward, rubber wood has been widely used in the production of plywood, particleboard and medium density fibreboard [1]. Recently a number of studies have been reported on the modification of wood. Several studies have been done on the modification of rubber wood such as flame retardant [2], termite resistant [3] and photostability [4]. Devi et al. (2002) had reported that impregnated rubber wood with polymer will enhance its mechanical properties (hardness, tensile strength and bending).

Previous researcher had proposed three types of magnetic wood which are impregnated type, powder type and coating type. Magnetic wood has been previously produced by impregnating magnetic fluid directly into the wood. Thus, it will maintain the woody texture and it is reported this process is the easiest among the three processes. Magnetic wood can be used as a heating boards, attraction boards and wave absorbers [5].

Woods are hygroscopic and hydrophilic materials. The moisture exists in wood with three phases, i.e., bound water, liquid water, and water vapor [6]. The movement of liquid water into wood may be driven by capillary action or water potential. Previous study suggested that aqueous solutions may flow through cell walls by way of cell wall

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capillaries and cell lumen [7, 8]. Previous study reported the application of Fick's second law (unsteady-state method) was used to investigate the absorption of moisture in wood fiber-based composites. This experiment does not involve bulk flow and in the absence of chemical reaction, which makes Fick's second law can be used to solve the problems of unsteady-state diffusion [9].

EXPERIMENTAL

Materials

Rubber wood samples were obtained from Forest Research Institute of Malaysia (FRIM) with dimensions of 50 x 25 x 15 mm. All the wood samples were oven dried at 105 ^{o}C for 24 hours to obtain weight (O.D.) w_i.. Ferrous chloride tetrahydrate ($FeCl_2 \bullet 4H_2O$), ferric chloride ($FeCl_3$), and sodium hydroxide (NaOH) were procured commercially and used without further purification.

Water Sorption

Prior to the impregnation of Fe_3O_4 into the rubber wood, the diffusion of water into the rubber wood was investigated. This is important because water acts as carrier of $Fe^{2+}$and Fe^{3+} for penetrating into internal structure of wood. A wood sample was immersed in a beaker containing 150 ml of distilled water. Load was applied on top of the sample to avoid flotation. In time interval the sample was taken out and wiped with filter paper to remove excess water on the wood surface before measuring the sample's weight (w_t). The soaking process was continued until the reading of the weight reached a plateau. The effect of temperature on the water sorption process was also investigated by placing the desicator into an oven at different temperatures (20, 40, and 60°C). Three samples were used for each temperature to obtain the average reading. The amount of water sorption was calculated using equation below:

$$M_t = \left(\frac{w_t - w_i}{w_i} \right) \text{x } 100\%$$

The diffusion coefficient of the water sorption process into rubber wood was evaluated using Fick's second law (Eq.1):

$$\frac{M_t}{M_e} = 1 - \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(\frac{-(2n+1)^2 \pi^2 Dt}{l^2} \right) \qquad (1)$$

Eq. 1 could be simplified to Eq. 2

$$\frac{M_t}{M_e} = \frac{4}{L} \left(\frac{Dt}{\pi} \right)^{1/2} \qquad (2)$$

where M_t is the mass uptake at time t and M_e is the mass uptake at equilibrium. From

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Eq.2 a graph was plotted of M_t against $t^{1/2}$. The diffusion coefficient (D) was calculated from the slope of the linear portion of the sorption curve [10]:

$$D = \frac{\pi L^2}{4}\left[\frac{E_2 - E_1}{\sqrt{t^2} - t^1}\right]^2 \qquad (3)$$

where
E = $(W-W_1)/(W_E-W_1)$
W = specimen weight (g)
W_1 = initial specimen weight (g)
W_E = specimen weight at equilibrium (g)
t = time (min)
L = half specimen thickness (mm)

2.3 *In situ precipitation of Fe_3O_4 into rubber wood*

Aqueous solutions of Fe^{2+} and Fe^{3+} were prepared by dissolving $FeCl_2 \bullet 4H_2O$ and $FeCl_3$ into deoxygenated distilled water at pH~2 separately. This is to avoid the formation of iron oxide precipitates [11]. Both solutions were mixed in a beaker at a molar ratio of Fe^{2+}:Fe^{3+} equals to 1:2. The beaker was then placed in a desicator and vacuum was drawn for a certain period. After that, the system was vented to atmospheric pressure and NaOH was added. The desicator was then placed into an oven for 24 h at desired temperature. After that, the sample was rinsed using distilled water and dried at 105˚C for 24 h in oven.

CHARACTERIZATIONS

The degree loading of Fe_3O_4 in the rubber wood sample was calculated using equation below:

$$\text{loading degree (\%)} = \frac{\text{final weight (g)} - \text{initial weight (g)}}{\text{initial weight (g)}} \times 100\%$$

The particle distribution of Fe_3O_4 loaded in the rubber wood block was determined using Atomic Absorption Spectrometer (AAS) 3300 Perkin Elmer. Two pieces samples (approximately 1 g each) were cut from 5 zones of the rubber wood for obtaining average reading. All the samples were soaked separately in 0.5 M HCl for 24 hours to dissolve the loaded Fe_3O_4. Then the solution was filtered with membrane filter (0.45 µm) and the concentration of Fe ions in the solution was analyzed using AAS.

The thermal properties of the magnetic wood were characterized using a thermogravimetric analyzer (Pelkin Elmer). The sample was heated from room temperature until 780˚C under nitrogen atmosphere. The activation energy (E_a) was calculated from the TG curve using Broido method. The Broido equation, as shown below, was used to calculate the kinetic parameters for the second stage of the thermal degradation of the magnetic rubber wood block:

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$$\ln\left(\ln\frac{1}{y}\right) = -\frac{Ea}{R}\frac{1}{T} + \ln\left(\frac{R}{Ea}\frac{Z}{R_H}T^2 m\right) \qquad (4)$$

The cross section of the magnetic rubber wood was examined by using a Leo 1450 VP model scanning electron microscope (SEM). The magnetic rubber wood sample was cross cut and coated with gold for the test.

The magnetisation and coercivity of the magnetic rubber wood block were measured using the LakeShore vibrating sample magnetometer (VSM) at room temperature. The sample of approximately 20mg was vibrated vertically and the dipole field of the sample induces an AC signal proportional to the magnetisation of the sample in a pair of coils.

RESULTS AND DISCUSSIONS

Water sorption

Figure 1 depicted that the water sorption into rubber wood was increased with temperature. The maximum water sorption was increased from 58.18 % to 80.72 % when temperature increased from 20 ^{o}C to 60 ^{o}C. High water sorption into the rubber wood at elevated temperature was attributed to the swelling of wood cell wall. This would result in higher number of hydroxyl group available for water sorption [4, 12]. The diffusion coefficient (*D*) of the water sorption process at various temperatures was calculated and given in Table 1. The *D* value was increased with temperature. Previous study reported that a change in temperature would result in pressure redistribution in the wood structure and enhanced the diffusion of ions [13]

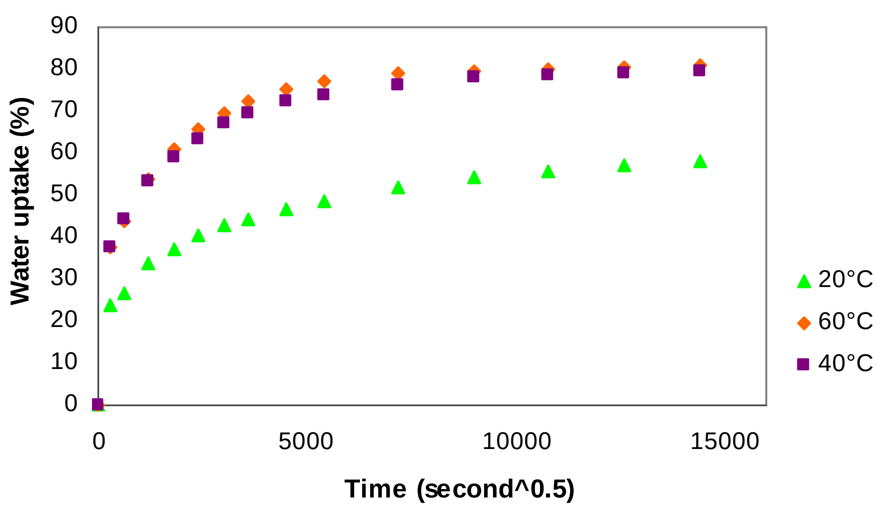

Figure 1: Water sorption into rubber wood at different temperatures.

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Table 1: Water sorption into rubber wood at different temperatures

Temperature (^{o}C)	M_e (%)	D 10^{-9} (m^2/s)
20	58.18	2.25
40	79.32	2.29
60	80.72	2.55

Degree of loading of magnetic wood

Figure 2 illustrated the effect of various concentrations of Fe on the loading degree of magnetic wood. The degree of loading was increased from 5.22 % to 20.05 % when the Fe concentration increased from 0.2 to 1.0 M. Higher concentration of Fe before the addition of NaOH had promoted the penetration of Fe ions into the wood structure. It could be attributed by the higher concentration gradient between the internal structure of the wood and the external suspension. Therefore more Fe_3O_4 nanoparticles would precipitate and subsequently deposited on the cell wall of the wood after the addition of NaOH.

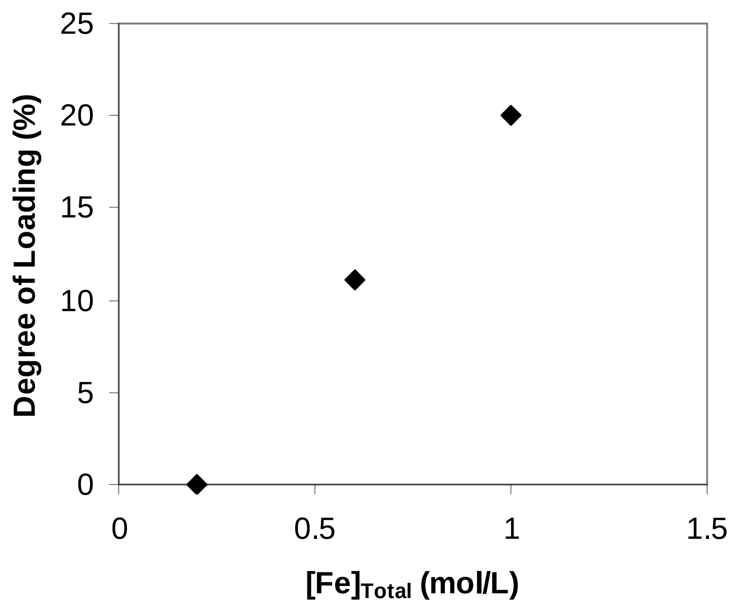

Figure 2: Variation of loading degree of magnetic rubber wood as a function of Fe concentration

Atomic absorption spectrometer (AAS)

Figure 3 demonstrated the profile of the Fe_3O_4 nanoparticles loaded into the rubber wood contains 11.1% of loading degree. This figure shows an unbalance of magnetic particle distribution in the rubber wood with higher degree of loading at the edge of the rubber wood than that of at the middle of the wood. This phenomenon was probably associated to the inhomogeneous of wood structure [5].

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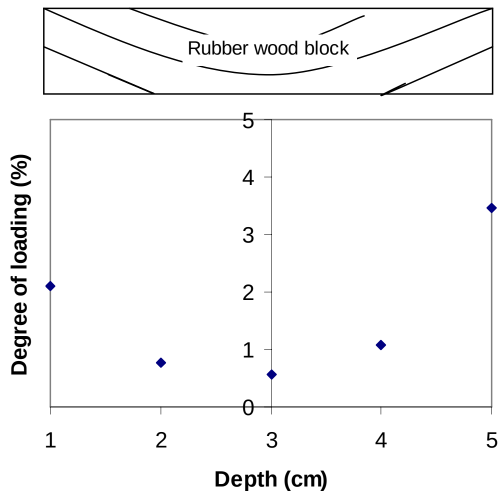

Figure 3: Graph shows degree of loading (g) vs depth of wood (cm)

Thermogravimetric analysis (TGA)

Figure 4 demonstrates the plots of ln (ln1/y) against (1/T) for the second stage of the thermal degradation. The slope and the intercept of the straight lines from the graph were used to calculate the activation energy (Ea). The activation energy of the unloaded rubber wood block was 81.106 kJ/mol and for the magnetic rubber wood which was loaded at 0.05 Fe^{2+} (20.05%) and 0.03 Fe^{2+} (11.10%) was found to be 77.811 kJ/mol and 45.408 kJ/mol respectively. The decreasing in the activation energy at 0.05 Fe^{2+} concentration was found to be due to the presence of inorganic salt (NaCl) and compound (Fe_3O_4) has catalyzed the decomposition process, thus, resulting in lower Ea [11].

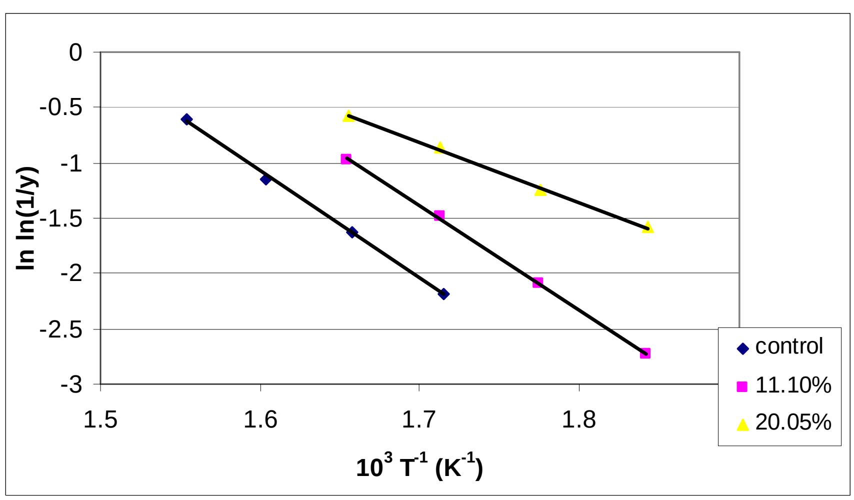

Figure 4: Plots of ln(ln1/y) vs. (1/T)

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Scanning electron micrographs (SEM)

Figure 5 illustrates the SEM micrographs of the rubber wood block which had been loaded with Fe_3O_4 nanoparticles at room temperature. These micrographs showed that Fe_3O_4 nanoparticles had been successfully deposited into the rubber wood block and the outcome had been confirmed by EDX. The average of the deposited Fe_3O_4 was 7.29 wt%.

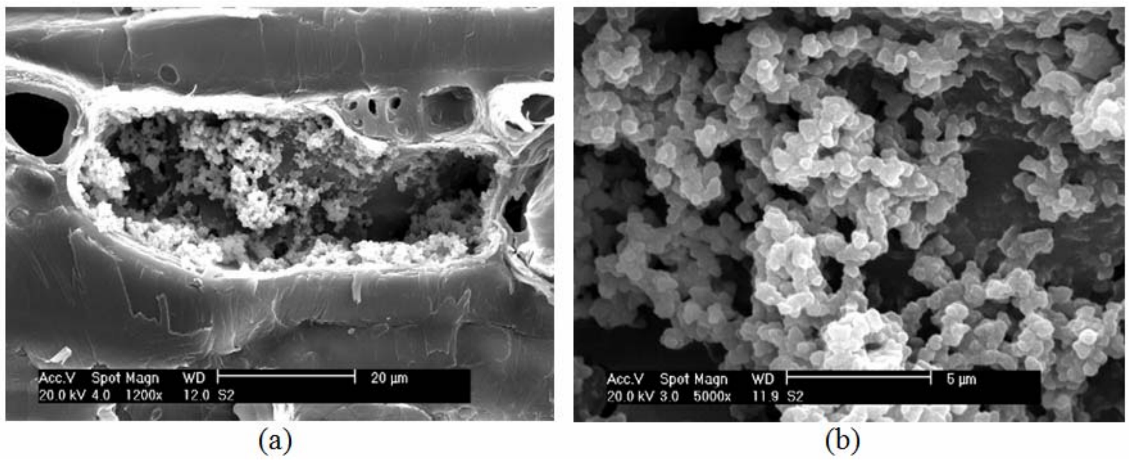

Figure 5: (a) and (b) SEM of treated magnetic rubber wood

Magnetic properties

The saturation magnetization of (*Ms*) is 52.56 emu/g and the coercivity (*Hc*) is 18.46 *Oe*. The results indicated that the rubber wood block that has been loaded with Fe_3O_4 nanoparticles possess magnetic properties. It is assumed that magnetic rubber wood block which was produced at higher concentration possess greater magnetic properties. It had been reported that the higher degree of loading the greater the *Ms* and *Hc* [11].

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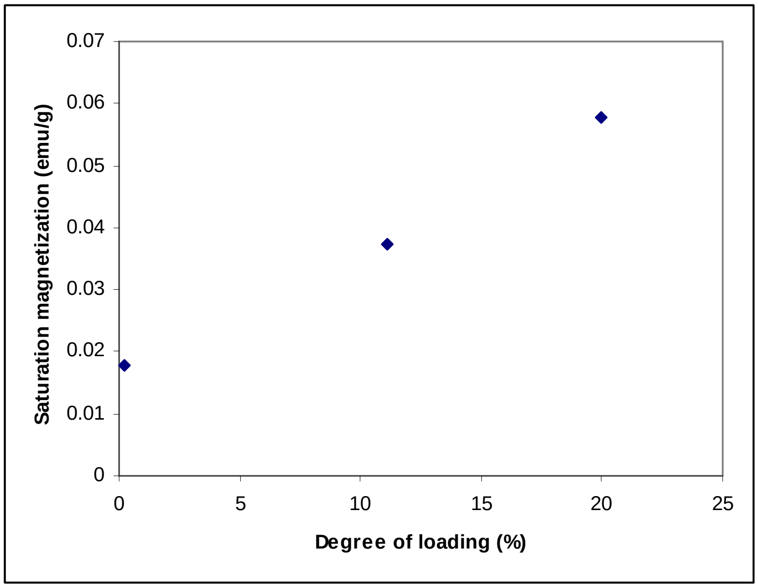

Figure 6: Graph of *Ms* (emu/g) vs. degree of loading (%)

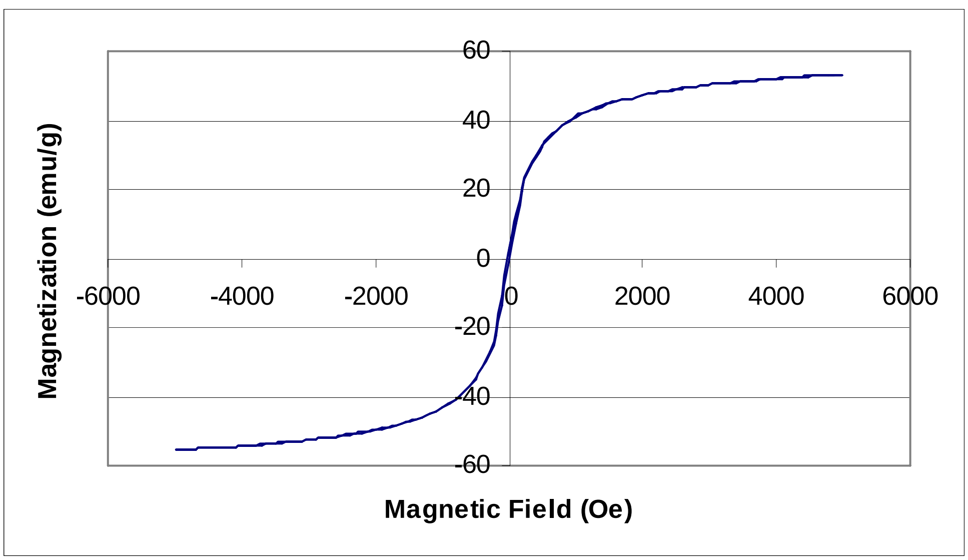

Figure 7: Hysteresis loop of *Ms* (emu/g) vs. *Hc* (kOe)

CONCLUSION

It can be concluded that water sorption onto rubber wood block was much more efficient at higher temperature (60˚C) than at 20˚C. Magnetic rubber wood block which was produced at room temperature gives a huge amount of Fe_3O_4 loaded into the rubber wood block.

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