Lignocellulose–Polymer Composite. III

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SYNOPSIS

A linear relationship was achieved between the polymer load and the monomer concentration up to 200% when china clay or talc replaced the glass in the initiating system, sodium bisulfite–soda lime glass, for the free-radical graft polymerization reactions using semi-chemical pulp of bagasse as substrate. The results showed that china clay is better than talc, which may be contributed to the difference in their network structure. The properties of the composites prepared from the cografted semichemical pulp–polymethyl methacrylate revealed that the china clay leads to composites with high compression strength and hardness. Deformation percent increased with increasing polymer load. However, decreasing or increasing the polymer load affects the properties of the composites up to a limit, where there is a maximum or minimum for both compression strength and hardness at china clay ratio of 2 or 3. Composites were also prepared from poly(methyl methacrylate)-cografted-pith of bagasse using the initiating system sodium bisulfite in the absence or presence of soda lime glass. Compression strength, deformation percent, and hardness increased on decreasing the glass ratio from 1 to 0, at nearly the same polymer load. The presence of waxes and resins decreased the compression strength of the composites prepared by impregnation of the lignocellulose in polymer solution. The hardness of these composites increased on removing waxes and resins. Removal of part of hemicellulose by alkali treatment of the lignocellulose has increased the effect on hardness. Alkali treatments of the substrates lead to a high deformation percentage. The compression strength of alkali-treated lignocellulose are lower than the untreated ones. The change of compression strength to deformation percent and the compressibility due to complete removal of waxes and resins by the extraction with methanol–benzene or partial removal of the waxes, resins, and hemicellulose through alkali treatment followed the change of both the compression strength and percent of deformation. Water uptake of the composites prepared in this work was ranged between 6.8 and 7%. After 48 h the water uptake increased to the range 8.5–14.1%. Impregnation of the composites in water for 72 h increased the water uptake to the range 10.2–18.1%. © 1993 John Wiley & Sons, Inc.

INTRODUCTION

In Part I of this series,[1] the properties of the lignocellulose–poly(ethyl methacrylate) composites were found to be affected by both the polymer load and the mesh size of the ground bagasse. The glass affects the properties of the composite. Replacing the glass with cupric oxide improved some properties of the composites while the use of ferric oxide instead of glass deteriorated their properties. The mesh size of the ground bagasse affects the properties of the composite.

In Part II of this series,[2] it was found that the change of the properties of lignocellulose–poly(methyl methacrylate) composites with polymer load is affected by the glass to lignocellulose ratio, and there is a limit for such effect. Ball-milling of the lignocellulose deteriorated the properties of the composites. Composite prepared by using sodium bisulfite–soda lime glass as initiator for grafting poly(methyl methacrylate) showed, in general, improved properties compared with ceric ammonium sulfate. The reverse was achieved on prehydrolyzing the substrate, which leads to inferior properties of the composite made.

Alkaline pretreatment of ground bagasse or semichemical pulp depressed the compression strength.

The other properties improved for composites from bagasse–semichemical pulp while they improved or deteriorated for samples from ground bagasse, depending on the kind and concentration of the alkali used in pretreatment and also on the particle size of bagasse. Subsequent mechanical treatment of the alkali-treated unground bagasse resulted in improved properties compared to those due to mechanical nontreatment of the ground one.[2]

In the current study, the performance of various cellulosic materials reinforcement in thermoplastics [e.g., polyethylene (PE) and polystyrene (PS)] has been compared by evaluating the mechanical properties of the composite materials. Experimental results indicate that the performance of three different celulosic materials are not the same.[3]

Wright and Mathias[4] prepared wood–polymer composite from southern pine and balsa wood by impregnating whole wood samples with comonomer solutions. The monomers were polymerized *in situ* using thermal activation or a combination of thermal and microwave cure. The same investigators[5] have also prepared ultralight wood–polymer composites by impregnating wood with comonomers solutions of ethyl (α-hydroxy methyl acrylate) and styrene and *in situ* polymerization. Various impregnation methods and cure conditions were compared. Initial findings confirm significant improvements in dimensional stability and mechanical properties.

In this part of the study, fillers such as china clay, talc, and glass were compared regarding their effects on the grafting reaction and composite properties. The effect of waxes and resins of the lignocellulose as well as the hemicellulose on the properties of the composite was investigated.

Properties of the prepared composites were studied to trace the influence of the composition of the substrates on these properties. Composites prepared from grafted pith bagasses and bagasse were compared.

EXPERIMENTAL

Substrates used were bagasse (mesh size 40) and pith of bagasse (mesh size 40). Polymers used were poly(methyl methacrylate) and polystyrene. The monomer used was methyl methacrylate.

Preparation of the Samples for Composites

The samples used for the preparation of composites were carried out by one of the following methods:

1. Graft polymerization
2. Impregnating the substrates in polymer solution.

Grafting was carried out by using sodium bisulfite–soda lime glass as initiator.[1] In some experiments, the glass was either omitted or replaced with china clay or talc. In these experiments 10 g of the substrates were placed in a 500-mL well-stoppered ground joint glass bottle; 0.3 g sodium bisulfite was added in a ratio to lignocellulose 3 : 100 at glass, clay, or talc to lignocellulose ratio 1 : 2 or 1 : 3, liquor to total solid ratio 10 : 1, and liquor to lignocellulose ratio 15 : 1. Monomer to lignocellulose ratio depends on the obtained polymer loads. The reaction bottle was placed in a water bath at 40°C and occasionally shaken for 20 h. The product was then thoroughly washed with water, left to dry at 60°C, and then weighed for the determination of the crude grafting yield:

$$\text{crude grafting yield: } C\% = [(F - S) - Z]/Z$$

where S is the weight of the glass, china clay, or talc, F the weight of the substrate, and Z the weight of the crude grafted product.

By Impregnation into Polymer Solutions

Two grams of the polymer was placed into a 500-mL glass beaker to which 200 mL of the proper solvent was added, e.g., benzene in the case of polystyrene and acetone in the case of poly(methyl methacrylate). Ten grams of the substrate were impregnated in the polymer solution and kept covered for 24 h at room temperature, and then the solvent was evaporated, dried, and weighed.

For preparing the composites. About 10 g of the product were heated at 170°C in a disk form (mold), and then pressed under 160 kg/cm², for 10 min. The resulting composite samples were investigated for:

1. Compression strength: determined as the weight at which the composite sample is deformed or breaks (ton/cm²).
2. Percent deformation: calculated from the difference between the length before and after compression divided by the first length × 100.
3. Hardness: measured by a hardness testing machine HPK by Ball Kögel, Leipzig.
4. Compression strength to percent deformation: calculated by the Poisson ratio obtained for the composite (Ref. 6, p. 127).

5. Compressibility K: calculated according to the following equation (Ref. 6, p. 30):

$$K = -(1/V)/(dV/dP)$$

where V is the volume of the composites and P is the pressure.

6. Water uptake was measured as mentioned in Part I[1] after 24, 48, and 72 h.

RESULTS AND DISCUSSION

Graft Polymerization by Using China Clay or Talc

Grafting reactions of poly(methyl methacrylate) onto lignocellulose by using china clay or talc instead of glass in the initiating system, sodium bisulfite–soda lime glass, were investigated. Series of experiments were carried out using different concentrations of methyl methacrylate monomer, namely, 30, 50, 100, 150, or 200%, based on the substrate used, which was semichemical pulp of bagasse.

The results illustrated in Figure 1 showed a linear relationship between the polymer load and the concentration of the monomer up to 200% in the presence of sodium bisulfite china clay or talc as initiating systems. From Figure 1, it is clear that clay is better than talc in the initiating system, which may be contributed to the difference in their network structure. It is worth mentioning that the mechanism of the initiation by these systems depends on
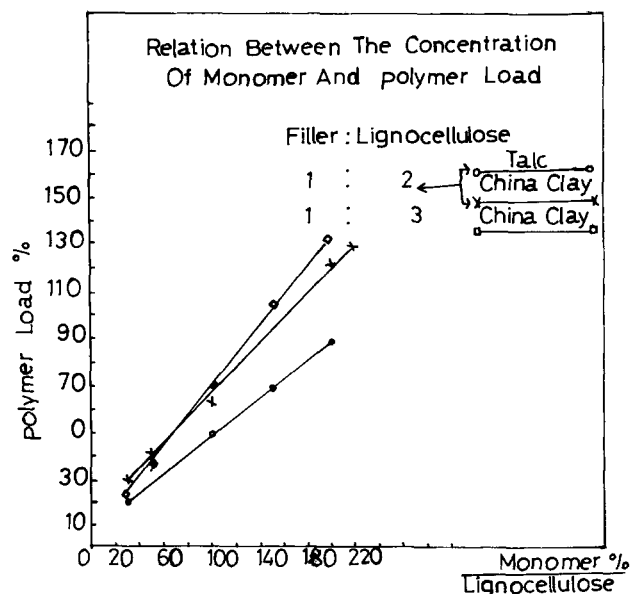

Figure 1   Sodium bisulfite 3 gm/100 gm lignocellulose. Liquor to lignocellulose ratio 15 : 1.

the trapping of the bisulfite radical inside the framework to form a so-called sulfur-impregnated solid.[1] Decreasing the ratio of the lignocellulose to china clay from 3 : 1 to 2 : 1 increased the percentage of polymer load at the same monomer to lignocellulose percentage up to 70%.

Regarding the properties of the composites prepared from lignocellulose-grafted poly(methyl methacrylate) using china clay or talc in the sodium bisulfite initiating system, it is clear from Table I that there is a maximum and minimum for the relations between polymer load and compression strength or between polymer load and hardness. Also, at china clay to lignocellulose 1 : 3 there is a maximum for the relation between the polymer load and compression strength or hardness, while, for the ratio 2, there is a minimum for these relations. When talc was used, the minimum was achieved for these relations and, at talc ratio 3 (Table I). It is also clear from Table I that the hardness of all composites prepared at china clay ratio 3 showed high values compared with those obtained at ratio 2. The use of china clay as filler in the lignocellulose–polymer composites showed high values of compression strength and hardness compared with composites prepared by using talc (Table I). Deformation percent increased with increasing polymer load while the compression strength to deformation percent decreased when china clay was used. On the other hand, there is a maximum in the ratio of compression strength at 68% polymer load when talc was used (Table I). Compressibility showed no relationship with polymer load when either talc or china clay was used (Table I). Water uptake decreased with increasing polymer load for both china clay and talc (Table I).

Composites from Bagasse and Pith of Bagasse

By Graft Polymerization

Bagasse of mesh size 40 and pith of bagasse of the same mesh size 40 were used as substrates for grafting poly(methyl methacrylate) in the presence and absence of soda lime glass in the initiating system sodium bisulfite. The grafting reactions were carried out on the treated and untreated substrates; their analyses are shown in Table II. The polymer load of the cografted polymers and the properties of the composites prepared therefrom are shown in Table III. It is clear from Table III that the polymer load was 6.64% when the substrate used was untreated pith of bagasse, and it becomes 8% for the methanol-benzene extracted of 0% waxes and resins (Table

Table I   Effect of Different Fillers Onto Composite Properties[a]

Type of Filler	Talc					China Clay									
Ratio of Filler	1 : 2					1 : 2					1 : 3				
Polymer Load (%)	20	41	48	68	88	28	36	62	116	120	23	38	68	105	132
Density (g/cm³)	1.15	1.07	1.22	1.01	1.29	1.52	1.42	1.38	1.17	1.35	1.36	7.42	1.37	1.35	1.31
Compression strength (tons/cm²)	1.08	1.06	1.46	1.04	1.79	1.25	1.28	1.89	1.19	1.99	2.1	2.28	2.1	2.08	2.06
Deformation (%)	43.9	32.1	42.9	3.3	36.4	14.0	19.0	22.3	24.8	30.9					
Compression strength to deformation	0.024	0.033	0.027	0.306	0.049	0.108	0.074	0.061	0.048	0.043					
Compressibility (K)	0.121	0.064	0.062	0.068	0.158	0.024	0.033	0.014	0.081	0.064					
Hardness (HPK)	698	671	725	665	964	879	936	984	720	844	915	98	1225	1100	1138
Water uptake % after															
24 h	17.8	15.7	12.4	7.6	5.7	17.2	16.9	18.8	6.4	3.6	9.8	7.8	6.5	4.5	2.7
48 h	19.8	27.8	24.3	15.8	12.2	17.8	28.4	12.1	8.2	3.9	13.3	13.6	9.4	7.7	4.6
72 h	22.1	32.6	23.4	15.0	8.4	19.9	30.3	24.8	9.1	4.96	23.4	18.4	11.8	8.0	5.6
7 days	24.9	29.0	36.3	10.72	10.2	25.2	35.0	15.5	10.5	8.1	19.5	19.8	16.2	7.6	7.0

[a] The lignocellulose used was semichemical pulp of bagasse; the monomer was methyl methacylate. Sodium bisulfite of 0.3% was used as the initiator. Temperature uses was 40°C.

II). The properties of the composite prepared from untreated pith of bagasse are significantly low compared with that prepared from the extracted one (Table III). The same deterioration is achieved for the sample prepared at 0% polymer load (Table III).

The graft polymerization reaction carried out by using extracted pith of bagasse and in absence of glass lead to 9.4 polymer load (Table III). The properties of the prepared composites from this substrate showed high compression strength and hardness compared with the composite prepared from the cografted substrate in the presence of glass ratio 2 and having 8% polymer load. Thus, the presence of glass has significant influence on the properties of the composites; such a result has been previously achieved using other substrates.[1] However, the absence of glass showed a slight increase in compression strength, while the hardness remained unaffected (Table III) for the composites prepared from both bagasse and pith of bagasse extracted with

Table II   Analysis of the Lignocelluloses

Material	Semichemical Pulp of Bagasse			Pith of Bagasse			Bagasse		
Type of Treatment Experiment	Methanol Benzene	2% NaOH	4% NaOH	Methanol Benzene	2% NaOH	4% NaOH	Methanol Benzene	2% NaOH	4% NaOH
Lignin content (%)	1.89	2.55	1.62	25.13	22.67	18.87	27.62	12.14	7.07
Ash content (%)	0.98	1.95	1.47	5.21	6.68	6.60	2.31	4.26	4.21
Extractable hemicellulose (%)	26.94	11.52	8.88	30.63	20.92	19.37	23.69	13.50	10.50
Waxes and resins content (%)	0	2.68	3.16	0	0.89	1.27	0	1.67	2.06
Yield (%)	94.5	83.2	75.8	80.0	78.2	60.8	93.6	70.8	65.9

methanol–benzene mixture. At 2% polymer load and in the presence of glass, the compression strength and the hardness as well are highly increased. Deformation percentage of the composite prepared from grafted-untreated pith of bagasse of 6.64% polymer load was 33.3% higher than that obtained from the extracted pith of bagasse with polymer load from 8 to 0%, and, at the same glass ratio, the deformation percentage of the composites prepared from these substrates increased from 12.0 to 26.8%. At zero polymer load, it is shown that with a decrease of the glass ratio from 1 to 0, the deformation percentage of the composite increased from 26.8 to 39.1%. Also, at nearly the same polymer load, 8 and 9.4%, the deformation percentage increased from 12.0 to 33.3% on decreasing the glass ratio from 1 to 0. Thus, the decrease of the glass ratio in the composite increased the percentage of deformation (Table III). On the other hand, the presence of glass increased the compressibility from 0.033 to 0.042 for composites prepared from methanol–benzene-extracted pith of bagasse, and from 0.056 to 0.198 for composites prepared from bagasse extracted with methanol–benzene.

The extraction of pith of bagasse with 4% sodium hydroxide solution for 24 h increased the polymer load up to 14% on carrying grafting reaction using sodium bisulfite–soda lime glass as initiator. The monomer used was methyl methacrylate. The compression strength of the composite produced was 0.5 tons/cm² and the hardness was 178 (Table III).

By Impregnation

Preparation of the composites was also carried out using lignocellulose impregnated into polymer solution; e.g., polystyrene in benzene and poly(methyl methacrylate) in aceton. The results illustrated in Table IV showed that poly(methyl methacrylate)-impregnated lignocelluloses lead to composites of mainly better properties, namely, compression strength, hardness, and compressibility, than those composites prepared by using polystyrene-impregnated lignocelluloses. On the other hand, the deformation percentages are lower for the composites prepared by using polystyrene solution than those prepared by using poly(methyl methacrylate) solution.

The influence of waxes and resins on the properties of the composites were traced when they were prepared from pith of bagasse (mesh size 40) and bagasse (mesh size 40) extracted with methanol–benzene mixture for 8 h to obtain 0% of the waxes and resins (Table II), and those were compared with

Table III Composites from Polymer-Grafted Lignocelluloses

Materials	Glass to Lignocellulose	Polymer Load (%)	Specific Gravity (g/cm³)	Hardness (HPK)	Deformation (%)	Compression Strength (tons/cm²)	Compression to Deformation	Compression Ressibility K	Water Uptake 24 h	Water Uptake 48 h	Water Uptake 72 h	Water Uptake 7 Days
Pith of Bagasse	1 : 2	6.64	0.918	90	33.3	0.23	0.006	0.303	9.2	14.96	24.27	19.9
Pith extracted with 1 : 1 methanol benzene mixture	1 : 2	8.0	1.136	683	12.0	0.60	0.050	0.042	15.4	30.2	33.9	38.3
	1 : 2	0	0.993	90	26.8	0.29	0.010	0036	6.0	14.4	15.2	20.3
	0 : 1	9.4	1.272	905	33.3	2.17	0.065	0.033	16.5	18.8	25.4	26.3
	0 : 1	0	0.811	90	39.1	0.39	0.010	0.069				
Pith treated with 4% NaOH	1 : 2	14.0	1.192	178	39.1	0.50	0.012	0.167	5.1	8.8	10.6	11.7
Bagasse extracted with 1 : 1 methanol benzene mixture	1 : 2	0	0.434	90	—	0.11	—	—	—			
	0 : 1	0	0.761	90	44.6	0.76	0.017	0.096	10.6	11.7	10.4	21.8
	0 : 1	2.0	1.149	459	41.7	1.59	0.038	0.056	—			
	0 : 2	2.0	0.993	90	26.8	0.39	0.020	0.198				

Table IV   Composites from Lignocellulose-Impregnated Polymers

Material	Polymer Used for Impregnation	Specific Gravity (g/cm³)	Compression (tons/cm²)	Hardness (HPk)	Deformation (%)	Compressibility	Compression Strength to Deformation	Water Uptake			
								24 h	48 h	72 h	7 Days
Pith of Bagasse	Poly(methyl methacrylate)	1.310	1.03	748	22.2	0.046	0.042	9.0	10.5	11.0	15.0
	Polystyrene	1.020	0.52	665	8.7	0.031	0.068	7.6	8.9	10.5	18.1
Pith extracted with methanol-benzene mixture	Polystyrene	1.147	1.09	695	7.7	0.015	0.141	10.4	14.1	18.1	22.4
Pith extracted with 2% NaOH	Polystyrene	1.149	0.49	708	10.0	0.043	0.049	8.6	14.1	18.1	22.4
Pith extracted with 4% NaOH	Polystyrene	1.229	0.47	720	10.0	0.045	0.047	6.8	10.2	12.3	14.7
Semichemical pulp	Poly(methyl methacrylate)	1.162	0.71	319	50.5	0.149	0.014	7.0	12.7	14.7	17.1
	Polystyrene	1.150	1.02	678	30.2	0.063	0.033	—	—	—	—
Semichemical pulp extracted with 2% NaOH	Polystyrene	1.251	0.70	804	18.4	0.063	0.038	—	—	—	—
Semichemical extracted with 4% NaOH	Polystyrene	1.154	0.71	275	25.6	0.070	0.027	—	—	—	—
Bagasse	Poly(methyl methylacrylate)	1.44	0.35	253	14.5	0.089	0.51	8.8	10.3	12.1	16.1
	Polystyrene	1.093	0.54	675	11.4	0.074	0.62	9.7	14.1	20.5	12.0
Bagasse extracted with 1:1 methonal benzene	Polystyrene	1.179	—	726	—	—	1.17	—	—	—	—
Bagasse treated With 2% NaOH	Polystyrene	1.278	0.58	1098	13.6	0.037	0.79	10.7	11.7	14.5	15.9
With 4% NaOH	Polystyrene	1.223	0.47	778	15.8	0.044	0.75	—	—	—	—

the untreated. Regarding the hardness of the composites, it is clear from Table IV that the removal of waxes and resins "0%" lead to composites with slight increased values; namely, from 665 to 695 for the pith of bagasse and from 675 to 726 for bagasse, respectively. The deformation percentages were slightly decreased on removing waxes and resins with methanol–benzene mixture. The results achieved in Table IV showed also that the compression strength increased, while the compressibility and the deformation percentage decreased on removing the waxes and resins, which means that the properties of the composites are better in the absence of waxes and resins. Thus, it can be concluded from those results that the presence of waxes and resins in the lignocellulose weakens the bonding between the polymer and the fibers of lignocelluloses.

On the other hand, the partial removal of waxes and resins can be arrived at by using 2 or 4% sodium hydroxide solution. The resin contents of these alkali-treated bagasse and pith of bagasse are found in Table II. These results showed that the waxes and resins contents of the pith of bagasse are lower than those of bagasse. The treatments with 2% sodium hydroxide solutions lead to higher values of waxes and resins than those treated with 4% sodium hydroxide solution. The treated lignocelluloses were then impregnated into polymer solutions and pressed into composites. The compression strength of the treated bagasse and pith of bagasse with 2 or 4% sodium hydroxide solutions are, respectively, 0.79, 0.75, 0.49, and 0.47 tons/cm^2. This mean that the compression strengths of the treated pith of bagasse are lower than those of the treated bagasse (Table IV). Also, the compression strengths of the treated lignocelluloses with 4% sodium hydroxide solutions are lower than those treated with 2% solutions. In this respect, the partial removal of the hemicelluloses as a result of the treatments with sodium hydroxide solutions must be taken into consideration. Thus, treatments of lignocellulose with 4% sodium hydroxide solution removed part of hemicellulose higher than the use of 2% sodium (see Table II). Therefore, the small contents of the waxes and resins could not overcome the decreased contents of hemicelluloses. Thus the partial removal of hemicellulose from lignocellulose through alkali treatment affects the compression strength of the composites prepared therefrom. To prove such an effect, composite samples were prepared from semichemical pulp of bagasse and the 2% alkali-treated one, having hemicellulose contents of 24.49 and 11.52%, respectively. The compression strength of these composites are 1.02 and 0.70 tons/cm^2, respectively (Table IV).

A slight decrease of the hemicellulose content of bagasse semichemical pulp through the treatment with 4% sodium hydroxide solution, namely, 8.88% (Table II), leads to no change in the compression strength of the prepared composite, namely, 0.70 tons/cm^2 (Table IV). Treatment of bagasse, pith of bagasse, and semichemical pulp of bagasse with 2 or 4% sodium hydroxide solutions, which partially removes hemicellulose and waxes, affects hardness properties of the composites prepared; for example, the hardness increased from 675 to 1098 on using 2% sodium hydroxide for treating bagasse, from 678 to 804 for treating semichemical pulp of bagasse, and from 695 to 708 for treating pith of bagasse. On using 4% sodium hydroxide solution for the treatments of the above-mentioned lignocelluloses, the hardness of the composites decreased or slightly increased compared with those prepared from the 2% sodium hydroxide treatments or those prepared from the untreated ones, respectively (Table IV).

Summing up, the removal of waxes and resins alone through the extraction with methanol–benzene mixture has no or little influence on the hardness properties of the composites. Partial removal of the hemicelluloses has an increased effect on the hardness, but it must be taken into consideration that not only the partial removal of the hemicellulose, which has an increased effect on the hardness, but also the swelling to a certain limit which took place during the alkali treatment and then deswelling due to the washing of the alkali-treated lignocelluloses increased the compactness of the fibers[7] and hence the increased values of hardness. This view can be emphasized by the increased hardness values of the composites prepared from 2% alkali-treated lignocelluloses compared to those values of the 4% alkali-treated ones; otherwise the 4% alkali-treated lignocellulose must lead to composites of high hardness values (Table IV). It is noteworthy that the swelling of the lignocelluloses can be traced from the formation of other shapes of lignocellulose such as cellulose II, which can be indicated by the forked-peak of the X-ray diagram,[8] when the lignocelluloses are treated with 4% sodium hydroxide solutions (Figs. 2 and 3).

However, alkali treatment of pith of bagasse with 2 or 4% sodium hydroxide solutions lead to composites of high deformation percentages Table IV. Semichemical pulp of bagasse showed highly decreased deformation percentages of the composites prepared from the 2% alkali-treated one while a slight decrease when the substrates was treated with 4% sodium hydroxide solution (Table IV).

The change of compression strength to deformation percent and the compressibility due to com-

plete removal of waxes and resins through extraction with methanol benzene mixture or partial removal of waxes, resins, and hemicelluloses through alkali treatment followed the change of both the compression strength and the percent of deformation (Table IV).

Water uptake after 24 h ranged between 6.8 and 7% for all composites prepared from bagasse, pith of bagasse, and semichemical pulp of gagasse, untreated and extracted with methanol–benzene mixtures and those treated with 2 and 4% sodium hydroxide concentration. After 48 h, water uptake increased to the range 8.4–14.1%. Impregnating pieces of composites in water for 72 h increased the water uptake to the range 10.2–18.1%. During the impregnation of pieces of composites for 7 days, the water uptake becomes 14.7–22.4% (Table III). Thus, the results obtained for the change of the properties of composites prepared from impregnation of the lignocellulose into polymer solution as a result of alkali treatment are in agreement with the results obtained in the previous work,[2] which revealed the influence of the alkali treatments of lignocellulose onto graft polymerization reaction and hence the change of the properties of the composites prepared therefrom.
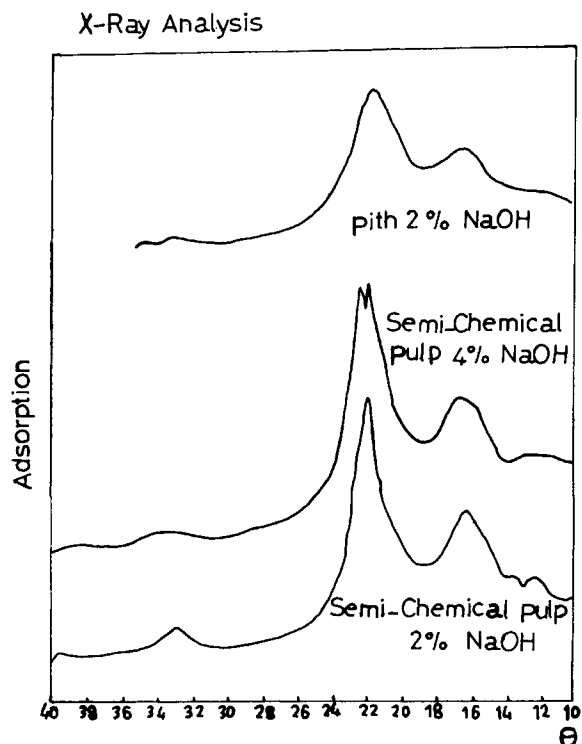
X-Ray Analysis

Figure 3   Lignocellulose to liquor 1 : 20; time of treatment 2 hrs.
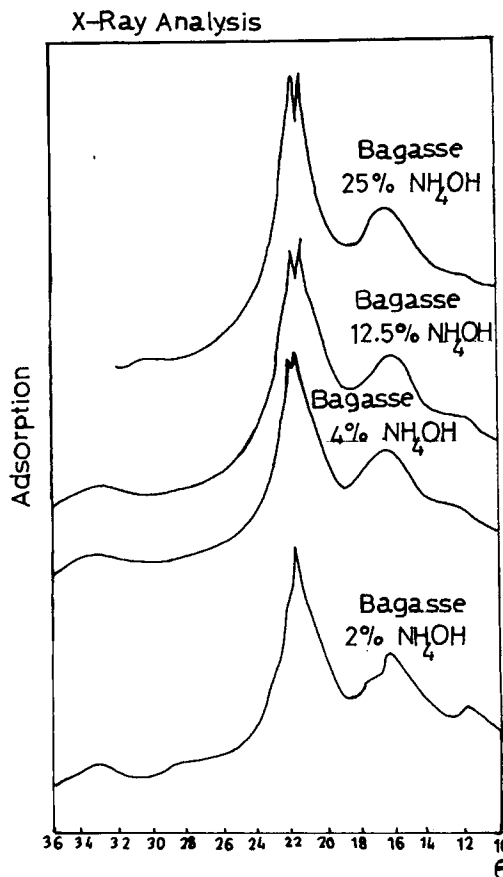
X-Ray Analysis

Figure 2   Lignocellulose to liquor 1 : 20; time of treatment 2 hrs.

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