

Fast Liquefaction of Bagasse in Ethylene Carbonate and Preparation of Epoxy Resin from the Liquefied Product

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ABSTRACT: Bagasse was rapidly liquefied in ethylene carbonate catalyzed by sulfuric acid at 140–170°C. The liquefaction was finished within 18 min. The liquefaction product was a mixture of polyols and phenolic compounds. The hydroxyl number of liquefied bagasse was in the range of 200–330 mg of KOH/g. The liquefied bagasse comprised two major fractions with different molecular weights. The average molecular weight of each fraction changed little during the liquefaction procedure, but the ratio of the con-

tent of the two fractions varied with time. An epoxy resin was prepared from the liquefied bagasse and diglycidyl ether of bisphenol A. The resin was cured with triethylene tetramine at 100°C. The resin presented higher adhesive shear strength and better thermal stability than a commercial epoxy resin. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 98: 1961–1968, 2005

Key words: blends; degradation; liquefaction; resins

INTRODUCTION

In recent years, the possibility of using lignocellulosic biomass as a new source for the production of chemicals and polymer materials has attracted great interest. However, because of the poor processing performance of lignocellulosic biomass, people seldom use plant biomass as a raw material directly. They have to convert lignocellulosic biomass into processable substances. A lot of conversion techniques have been proposed in the past few decades. Liquefaction is one of the useful and effective techniques.^{1,2}

The liquefaction of lignocellulosic biomass can be classified into two types based on the purposes and the reaction techniques. The first type is carried out at a high temperature and a high pressure and converts lignocellulosic materials into low-molecular-weight fuels and chemicals.^{3,4} The second type of liquefaction can be carried out under relatively mild conditions such as atmospheric pressure and a relatively low temperature and can convert biomass into polymer

materials or feedstock to manufacture polymer materials.

Liquefaction to convert plant biomass into polymer materials is usually conducted in organic solvents to prevent the recondensation of the degraded fragments of lignin and to increase the solubility of the liquefaction products. Polyhydric alcohols are commonly used solvents. Shiraiishi and coworkers^{5–7} successfully liquefied wood and wood waste in ethylene glycol, poly(ethylene glycol), and glycerin. The products have been used to manufacture polyurethane foams and epoxy resins.^{5,8–10} Many kinds of woods, chemically modified woods, wood wastes, and some nonwoods have been successfully liquefied with this method.^{6,11,12}

The use of polyhydric alcohols as liquefaction reagents, however, has the drawback of a low liquefaction speed. To enhance the liquefaction speed, Yamada and Ono¹³ adopted cyclic carbonates with high permittivity, such as ethylene carbonate (EC) and propylene carbonate, as the liquefaction reagents and liquefied bagasse rapidly. The rate of the EC liquefaction of cellulose and hardwood was approximately 10 times faster than that of polyhydric alcohol.

Epoxy resins are widely used as packaging and insulation materials and adhesives because of their excellent moisture, solvent, and chemical resistance and good adhesion to many substrates. However, they are usually used with some other materials in commercial applications. Lignin has a highly branched structure consisting of phenyl propane units. The con-

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tent of phenolic hydroxyls in the lignin macromolecules allows the utilization of lignin as a substitute for phenol in the synthesis of an epoxy resin. The introduction of the aromatic structure in lignin into the epoxy resin can enhance the adhesive strength and improve the thermal properties. Several approaches have been taken to incorporate lignin or lignin derivatives into epoxy resins,^{14–17} In the lignin molecules, a large number of the phenolic hydroxyls are etherified. Liquefaction can release some of the etherified hydroxyl groups and reduce the molecular weight of lignin. The release of hydroxyl groups can increase the number of reactive groups. The reactivity of lignin can thus be improved.⁹

To accelerate the liquefaction procedure and restrain the recondensation of the degraded fragments of lignin, we adopted EC as the liquefaction agent and sulfuric acid as the catalyst. The liquefaction of bagasse was finished within 18 min. The influence of the reaction conditions on the liquefaction of bagasse was investigated. The liquefied product was used as the polyol component in preparing an epoxy resin via the reaction with diglycidyl ether of bisphenol A (DGEBA). The adhesive shear strength of the obtained epoxy resin was evaluated. The thermal properties were also studied with differential scanning calorimetry (DSC) and thermogravimetry (TG).

EXPERIMENTAL

Materials

Bagasse was a byproduct of the Doumen Sugar Refinery (Guangdong, China). It was milled to 20–80 mesh and dried at 105°C overnight before use. EC was a commercial product of Fluka Co., Ltd. (Buchs, Switzerland). Other reagents were chemically pure or analytical-reagent-grade and were used as received.

Liquefaction procedure and evaluation of the residue content

Bagasse was added to a three-necked flask equipped with a reflux condenser and a stirrer. EC and sulfuric acid were added according to the predesired composition into the flask when the flask was heated to the desired temperature for liquefaction. After a defined duration, the reactor was rapidly cooled to room temperature with ice water. Then, the product was dissolved in a mixture of dioxane and water (4/1 v/v). The solution was filtered to isolate the residue. Then, the residue was rinsed thoroughly with dioxane and dried at 105°C for 24 h. The mass of the residue was determined, and the residue content was calculated with the following equation:

$$\text{Residue(\%)} = \frac{\text{Mass of residue}}{\text{Mass of starting bagasse}} \times 100\%$$

Each liquefaction run was repeated three times. The average data of the results were adopted.

Characterization of the liquefaction products

The acid number of the liquefaction product is defined as the number of milligrams of KOH required to neutralize the acid in 1 g of the liquefaction product. The acid number was measured according to Yao et al.'s method.⁵ About 1 g of the liquefaction product was diluted in a 4 : 1 dioxane/water solvent and stirred for 2 h. It was then titrated with a 0.1 mol/L KOH aqueous solution. The change in the pH was monitored by a PHS-3C pH meter (Rex Instrument Factory, Shanghai, China). The volume consumed at the neutral point was obtained from the neutralization curve. The acid number was calculated as follows:

$$\text{Acid number (mg of KOH/g)} = 56.1 \times V \times c/m$$

where V is the volume of the KOH solution consumed at the neutral point (mL), c is the concentration of the KOH solution (mol/L), and m is the mass of the liquefaction product (g).

The hydroxyl number of the liquefaction product is defined as the mass of KOH equivalent to that of the phthalic anhydride consumed in the phthalification of 1 g of the sample. The hydroxyl number was measured according to Yao et al.'s method.⁵ About 1 g of the liquefaction product was first esterified with 25 mL of a phthalic anhydride solution comprising 15% dioxane and 75% pyridine at 100°C for 2 h. Then, the extra phthalic anhydride was determined by back-titration with a 1 mol/L NaOH aqueous solution. The change in the pH was monitored by a PHS-3C pH meter. The volume of the NaOH solution consumed at the neutral point was obtained from the titration curve. The hydroxyl number was calculated according to the following equation:

$$\begin{aligned} \text{Hydroxyl number (mg of KOH/g)} \\ = 56.1 \times (V_1 - V_2) \times c/m + A \end{aligned}$$

where V_1 is the volume of the NaOH solution consumed in the blank titration, V_2 is the volume of the NaOH solution consumed in the back-titration (mL), c is the concentration of the NaOH solution (mol/L), m is the mass of the sample (g), and A is the acid number of the sample (mg of KOH/g).

The IR spectra of the residues and liquefaction products were recorded on a WQF-410 Fourier transform infrared spectrophotometer (KBr pellets (Beijing No. 2 Optical Instruments, Beijing, China).

The molecular weight and the molecular weight distribution of the product were determined with a Waters gel permeation chromatography (GPC) system

(Waters Corp. Milford, MA). The apparatus comprised a 515 pump, an Ultrastyrigel 500-Å column, and a 410 refractive-index detector. Tetrahydrofuran was used as the eluent. A series of narrow-distribution polystyrenes was used as a standard sample to calibrate the column. The column temperature was 40°C. The flow rate was 1 mL/min.

Preparation of the epoxy resin

The liquefied bagasse was mixed with DGEBA (epoxy equivalent = 227) in an earthenware mortar, and the mixture was left at room temperature for 24 h. Triethylene tetramine (TETA), the curing agent, was added to the mixture after that. Four types of resins were synthesized with different dosages of liquefied bagasse. The concentration of the curing agent varied in the range of 10–25 wt % of the total weight of the resins. The control experiment was conducted by the stoichiometric mixing of DGEBA and TETA without any addition of liquefied bagasse. The resins were cured at room temperature for 1 day and then cured at 100°C.

Characterization of the epoxy resin

Single lap shear joints fabricated with stainless steel substrates (125 × 25 × 2 mm) with an overlap length of 12.5 mm were used to evaluate the adhesive tensile shear strength of the cured resin. Hot pressing was conducted at 100°C under a pressure of 2.50 MPa for 1–3 h. The tensile adhesive shear strength was measured on a WE-100 universal testing machine (Guangzhou Testing Machine Factory, Guangzhou, China) at a crosshead speed of 2 mm/min according to Chinese National Standard GB7124-86.

DSC analysis of the cured resins was performed on a PerkinElmer Pyris Diamond Ta Lab System differential scanning calorimeter (PerkinElmer, Boston, MA). The samples were run at a constant heating rate of 10°C/min under a nitrogen atmosphere between 60 and 300°C.

Thermogravimetric analysis (TGA) of the cured resins was performed on a PerkinElmer PE-Pyris thermal analyzer at a heating rate of 10°C/min under a nitrogen atmosphere between 30 and 500°C.

RESULTS AND DISCUSSION

Liquefaction of bagasse

Cyclic carbonates have been used as effective solvents because of their high permittivity value. The permittivity of EC is 89.8.¹⁸ It is known that, for an acid-catalyzed reaction in nonaqueous solvents, the acid potential depends on the permittivity of the solvent. A relatively high permittivity can lead to a high acid

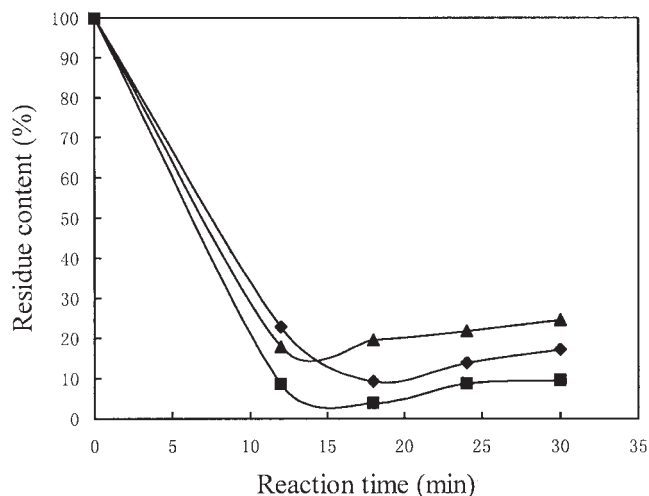


Figure 1 Effect of the temperature on the residue yields. The liquid ratio was 4, the catalyst concentration was 3%, and the temperatures were (◆) 140, (■) 150, and (▲) 160°C.

potential.¹³ For the acid-catalyzed liquefaction of lignocellulosic biomass, the use of EC can promote the reaction because of the high permittivity of EC.

Bagasse was found to be completely liquefied in EC within 18 min. The liquefaction rate of bagasse in EC was approximately 10 times faster than that in polyhydric alcohol, which lasted for 180 min, as reported in the literature.¹⁹

The influences of the reaction time, temperature, catalyst dosage, and liquid ratio on the liquefaction of bagasse were investigated.

Figure 1 shows the residue content in the liquefied bagasse versus time at different temperatures. The minimum residue content was obtained at 150°C. However, the residue content tended to increase when the temperature reached 160°C. The minimum residue content at this temperature was even higher than that at 140°C. The results indicate that temperature has a great influence on the liquefaction efficiency. It seems that enhancing the temperature promotes the degradation of plant fibers at a relatively low temperature and that it favors the recondensation of degraded fragments at a high temperature. At a low temperature, the fibrous raw materials decomposed slowly, so the residue content decreased at a low speed. When the temperature reached 150°C, the decomposition was accelerated, and the residue content was reduced drastically. However, a further increase in the temperature could promote the recondensation of the reaction intermediate, so the residue content tended to increase.

The effect of the liquefaction time on the residue content can also be observed in Figure 1. In each run of the experiment, the residue content decreased rapidly during the initial stage of the reaction; this indicates that the bagasse components decomposed and dis-

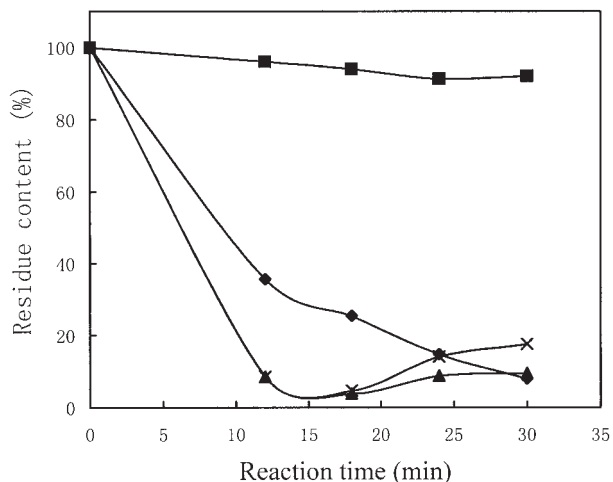


Figure 2 Effect of the catalyst dosage on the residue yields. The temperature was 150°C, the liquid ratio was 4, and the catalyst concentrations were (■) 0, (◆) 1, (▲) 3, and (×) 4%.

solved rapidly during this stage. The residue content tended to increase after 15 min of reaction, and this suggested that the recondensation of the degradation intermediates dominated the reaction at this time. It has been known that the free-radical fragments formed in the degradation of lignin easily condense to form char in the presence of an acid.²⁰ The content of the free-radical fragments formed in the degradation of bagasse increased with the increase in the reaction time. When the concentration of the degradation intermediates was high enough, recondensation occurred easily, and the residue content increased.^{20,21} Accordingly, the reaction time should not exceed 18 min.

Figure 2 shows the effect of the acid catalyst on the residue content. The residue content decreased drastically with an increase in the catalyst dosage during the initial stage of liquefaction; this shows that increasing the catalyst dosage can promote liquefaction during this stage. However, the variation of the residue content presented different regularities in the last stage when different amounts of the catalyst were used. The residue content began to increase when the catalyst content was in excess of 3%. However, no similar phenomenon was observed when the catalyst dosage was low. We have known that the degradation fragments of lignin easily recondense in an acidic medium.¹⁹ The recondensation of the free-radical intermediates can lead to an increase in the residue content. It can be concluded that an increase in the catalyst dosage accelerates not only the degradation reaction of macromolecules of lignocellulosics but also the condensation reaction of degraded fragments.

Figure 3 shows the effect of the liquid ratio on the residue content. The residue content was sensitive to the liquid ratio. The liquefaction efficiency was signif-

icantly improved by an increase in the liquid ratio. However, increasing the liquid ratio was not always effective in improving the liquefaction efficiency. When the liquid ratio exceeded 4, the residue content did not further decrease. This result can be interpreted from the functions of EC. EC, which has an excellent capability for donating protons, can block the free-radical intermediates that are formed during the liquefaction procedure and prevent the recondensation of the fragments. It can be converted into ethylene glycol after releasing carbon dioxide in the presence of an acid catalyst.²² It can also dissolve the degraded fragments. However, when the amount of EC is enough to dissolve the majority of liquefied bagasse, a further increase in the liquid ratio cannot continuously improve the liquefaction efficiency.

Structural characteristics of liquefied bagasse

Figure 4 presents the changes in the hydroxyl number and acid number of liquefaction products as functions of the reaction time. The hydroxyl number increased during the initial stage of liquefaction, whereas the acid number did not change; this indicated that the hydroxyl content increased during this stage and that the carboxyl content had no significant change. As an increase in the hydroxyl content is usually derived from the cleavage of the ether linkage of lignin and cellulose units, it can be concluded that the degradation of the macromolecules of plant fiber is the dominant reaction of liquefaction during the initial stage. The hydroxyl number and the acid number tended to decrease drastically at the reaction time of 20 min. The decrease can be explained as the oxidation of the hydroxyl groups and the esterification between the hydroxyl groups and carboxyl groups.

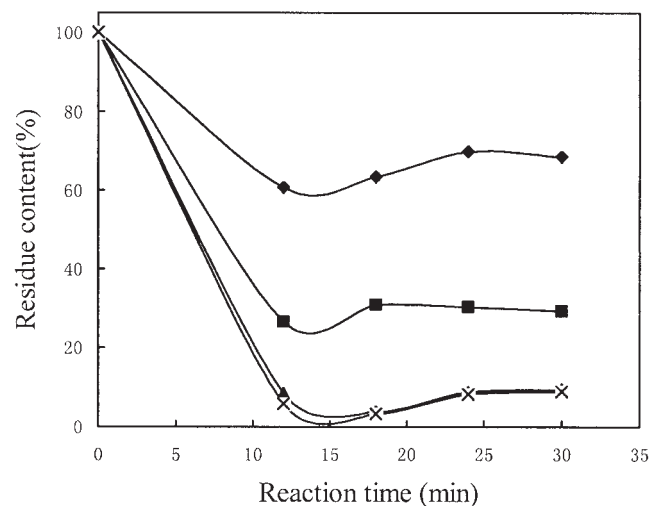


Figure 3 Effect of the liquid ratio on the residue yields. The temperature was 150°C, the catalyst concentration was 3%, and the liquid ratios were (◆) 2, (■) 3, (▲) 4, and (×) 5.

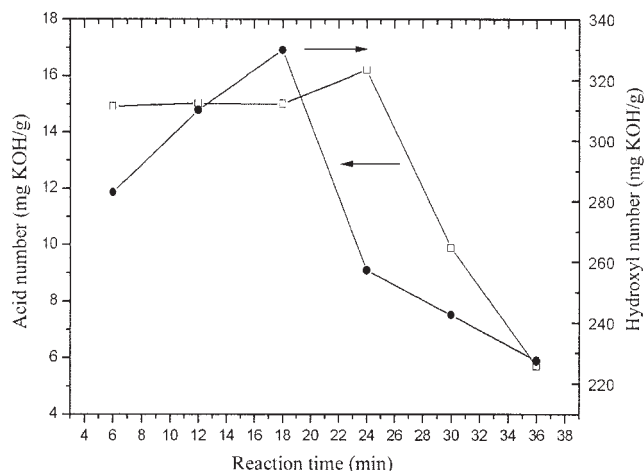


Figure 4 Hydroxyl number and acid number of the liquefaction mixture as functions of the reaction time. The temperature was 150°C, the liquid ratio was 4, and the catalyst concentration was 3%.

The IR spectrum of the liquefied bagasse is shown in Figure 5. The wide band at 3300–3500 cm^{-1} in Figure 5 represents the stretching of O—H. The duplicate bands at 1800 cm^{-1} represent the coincident stretching of C=O in the cyclic carbonate. The bands at 1608, 1510, and 1458 cm^{-1} represent the stretching of the aromatic rings of lignin. The band at 1116 cm^{-1} represents the stretching of C—O of cellulose. All these bands in Figure 5 indicate that the liquefaction product is a mixture of polyols and phenolic compounds.

The IR spectrum of bagasse and those of the residues obtained at different reaction times are shown in Figure 6. The IR spectrum of the residue formed after 6 min of reaction (curve 2) is similar to that of bagasse

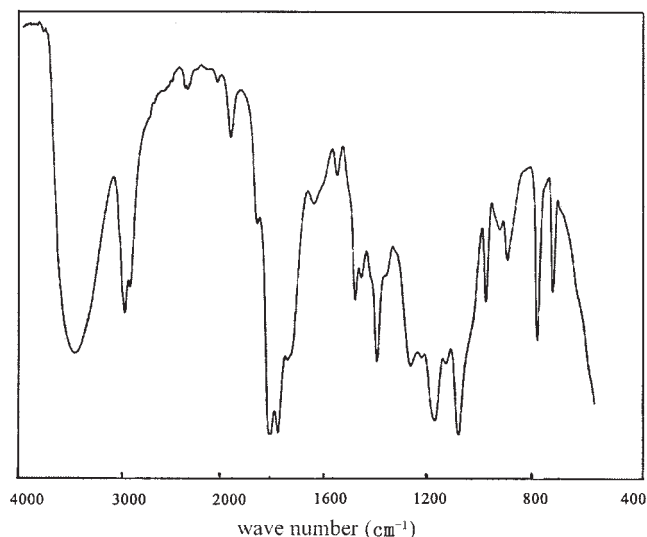


Figure 5 IR spectra of the product of liquefied bagasse. The temperature was 150°C, the liquid ratio was 4, the catalyst concentration was 3%, and the reaction time was 18 min.

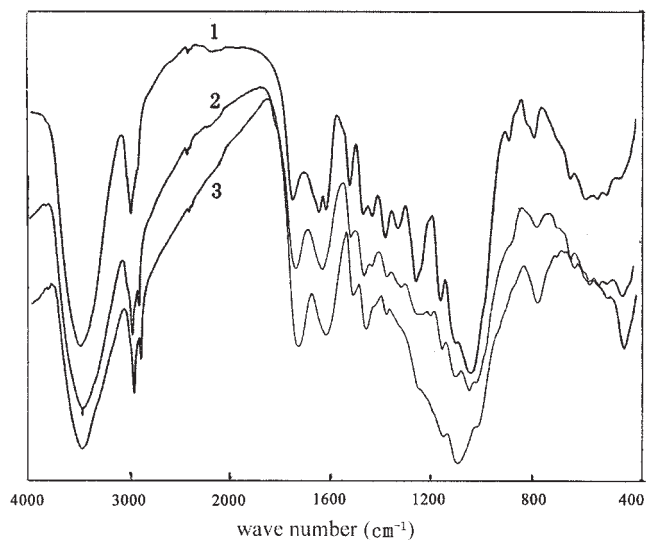


Figure 6 IR spectra of the liquefaction residue at different reaction times: (1) bagasse, (2) residue (6 min), and (3) residue (18 min). The temperature was 150°C, the liquid ratio was 4, and the catalyst concentration was 3%.

(curve 1). The bands at 1100–1400 cm^{-1} representing carbohydrates and lignin in curve 2 are weaker but can still be observed; this indicates that the liquefaction of bagasse was not completed. No bands can be observed at 1100–1400 cm^{-1} in the IR spectrum of the residue obtained after 18 min of reaction (curve 3). The spectrum indicates that the lignin and cellulose in bagasse were almost completely transferred into the liquid phase.

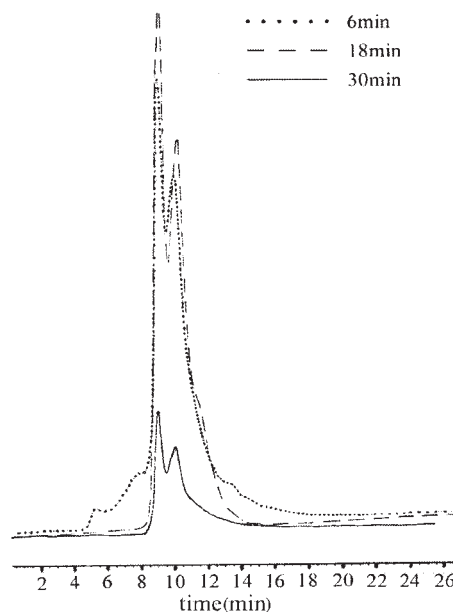


Figure 7 GPC diagrams of the liquefied bagasse obtained at different times.

TABLE I
Peak Area Ratio Between the Two Major Fractions
in GPC at Different Times

| Reaction time (min) | Peak area ratio |
|---------------------|-----------------|
| 6 | 0.63 |
| 12 | 0.48 |
| 18 | 0.55 |
| 24 | 0.62 |
| 30 | 0.72 |

The GPC diagrams of the three liquefaction products obtained at different times are shown in Figure 7. Each liquefaction product comprised two major peaks representing two major fractions having different molecular weights. The weight-average molecular weights (M_w 's) of the high-molecular-weight fraction obtained at 6, 12, and 18 min were 1406, 1453 and 1425, respectively, whereas the number-average molecular weights (M_n 's) were 1330, 1385, and 1383, respectively. The M_w values of the low-molecular-weight fraction obtained at 6, 12, and 18 min were 760, 779, and 778, respectively, whereas the M_n values were 750, 765, and 765, respectively. The polydispersity of each fraction varied between 1.013 and 1.057. The data indicate that each fraction was almost monodisperse. Considering the errors in the molecular weight measurement, we conclude that the reaction time has little influence on the molecular weight of the liquefaction product.

The IR spectrum of the high-molecular-weight fraction presented bands at 1609, 1510, and 1549 cm^{-1} , demonstrating that this fraction is an aromatic substance. It is evident that the high-molecular-weight fraction is derived from lignin in the bagasse. The IR spectrum of the low-molecular-weight fraction presented adsorption at 1455, 1162, 1116, and 1000 cm^{-1} , which are typical bands of cellulose. No bands representing aromatic compounds were observed. It is concluded that the low-molecular-weight fraction was derived from carbohydrate.

The GPC curves obtained at different reaction times had different shapes indicating that the molecular weight distribution was changed during the liquefaction procedure. The peak area ratios between the high-molecular-weight fraction and the low-molecular-weight reaction of the liquefied bagasse obtained at different reaction time are shown in Table I. The data demonstrate that the peak area ratio between the two major fractions was changed during the reaction. The content of the high-molecular-weight fraction decreased with time in the early stage of liquefaction. After that, the content of the high-molecular-weight fraction tended to increase. The result indicates that the degradation of bagasse, which increases in the content of the low-molecular-weight fraction, is the dominant reaction in the early stage of liquefaction. With an increase in the reaction time, the concentra-

tion of the degradation intermediates increased, and the possibility of the recondensation of degraded fragments increased; this enhanced the content of the high-molecular-weight fraction.

Preparation and properties of epoxy resins

Epoxy resins usually present excellent adhesive performance. In normal cases, however, epoxy resins are rarely used without the incorporation of other materials. Filling and polyblending are sometimes used to provide additional mechanical properties or to modify the physical characteristics of the blends. The performances of the epoxy resin are therefore improved. Feldman et al.¹⁴ manufactured an epoxy adhesive by polyblending bisphenol A with kraft lignin. A very complex matrix from a set of linkages between lignin units was introduced into the epoxy resin. The adhesive shear strength was significantly enhanced when 20% lignin was polyblended.

In this study, the polyblends were prepared by the stirring of the epoxy prepolymer and liquefied bagasse. Then TETA, the curing agent, was added to the mixture. The resulting mixture was stirred for 5 min and cured at 100°C for 2 h.

Table II shows the effect of the dosage of liquefied bagasse on the adhesive shear strength of the thermally cured polyblends. The results show that the cured resin polyblended with liquefied bagasse presented much higher adhesive shear strength than the epoxy resin cured under the same conditions without liquefied bagasse. The adhesive shear strength of the samples glued with the polyblends had values ranging from 6.61 to 19.00 MPa. The highest adhesive shear strength, 19.00 MPa, was achieved when 25% liquefied bagasse was added. However, the adhesive shear strength began to decrease when the concentration of liquefied bagasse reached 30%. These results indicate that the presence of liquefied bagasse has a beneficial effect on the shear strength. The improved adhesive shear strength was probably the result of a crosslink between liquefied bagasse and the epoxy resin network.

TABLE II
Adhesive Shear Strength of the Cured Resins with
Different Dosages of Liquefied Bagasse

| Dosage of liquefied bagasse (%) | Curing agent content (%) | Curing temperature (°C) | Curing time (h) | Adhesive shear strength (MPa) |
|---------------------------------|--------------------------|-------------------------|-----------------|-------------------------------|
| 0 | 15 | 100 | 2 | 6.61 |
| 20 | 15 | 100 | 2 | 7.21 |
| 25 | 15 | 100 | 2 | 19.00 |
| 30 | 15 | 100 | 2 | 9.62 |

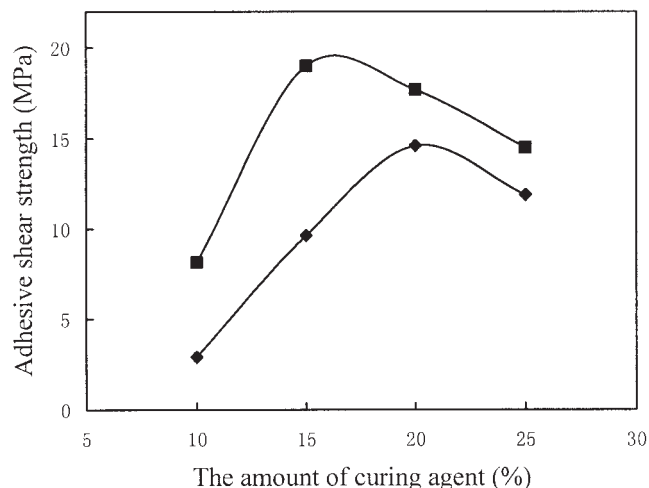


Figure 8 Adhesive shear strength of the cured resin versus the amount of the curing agent. The epoxy resin/liquefied bagasse ratios were (♦) 2 and (■) 3 (curing conditions: 2 h and 100°C).

Figure 8 shows the effect of the amount of the curing agent on the adhesive shear strength of the cured resin. An increase in the amount of the curing agent enhanced the adhesive shear strength drastically. The adhesive shear strength of the polyblends varied from 2.91 to 19 MPa when 10–25% of TETA was added. The adhesive shear strength increased with an increase in the TETA dosage. The highest adhesive shear strength, 19 MPa, was obtained when the TETA dosage was 15%. The adhesive shear strength, however, decreased drastically when the concentration of TETA reached 25%.

DSC diagrams of cured pure DGEBA resin and blends of DGEBA and liquefied bagasse are shown in Figure 9. The glass-transition temperature (T_g) of the

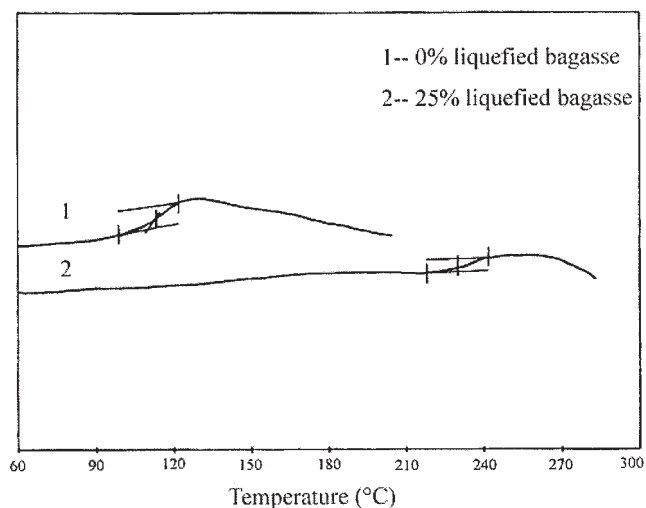


Figure 9 DSC diagrams of the cured epoxy resins (curing conditions: 15% TETA, 100°C, and 2 h).

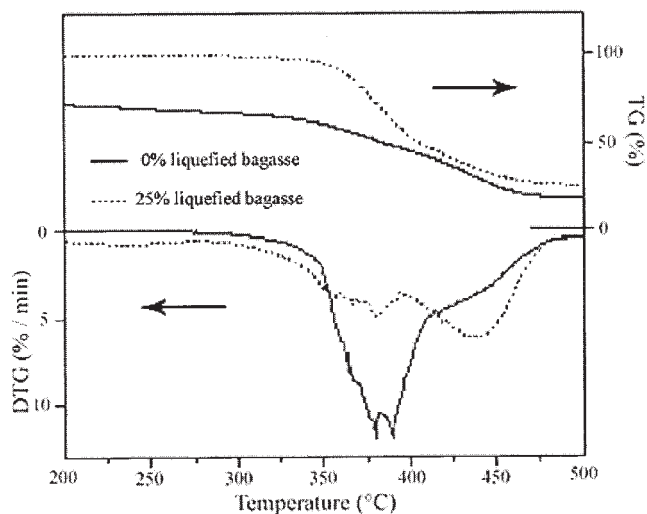


Figure 10 TGA diagrams of the cured resins (curing conditions: 15% TETA, 100°C, and 2 h).

cured resin polyblended with 25% liquefied bagasse was 230.41°C. In contrast, T_g of the pure epoxy resin cured under the same conditions was 111.19°C. The data indicate that T_g increased when the liquefied products were added to the epoxy resin. The changes in T_g can be attributed to the aromatic rings and the highly branched fragments degraded from lignin. A highly branched aromatic structure in the epoxy resins can enhance the rigidity of the molecular chains and restrict the motion of the molecular chains. The increase in T_g can enhance the thermal stability of the cured resin.

The pyrolysis behavior of the pure epoxy resin and the polyblends was investigated with TGA. Figure 10 shows the TG and differential thermogravimetry (DTG) curves of cured pure epoxy resins and cured polyblends. The TGA curve of the cured resin polyblended with 25% liquefied bagasse has two regions, indicating that the thermal degradation of the cured polyblends occurs in two steps. The lower decomposition temperature of the polyblend was 397.87°C, and the higher one was 437.19°C. The decomposition temperature of the pure epoxy resin was 392.75°C, and this indicates that the epoxy groups in the polyblend decomposed at a relatively low temperature. The rigid aromatic structure of lignin derivatives can restrict the motion of molecular chains and improve the thermal stability of epoxy–liquefaction product polyblends.

CONCLUSIONS

Bagasse can be rapidly liquefied in EC in the presence of sulfuric acid under atmospheric pressure. The liquefaction product is a mixture of polyols and phenolic compounds. The reaction can be finished within 18 min at 150°C. The temperature, catalyst content, and

liquid ratio have great influence on the residue content. Increasing the amount of the acid catalyst enhances the liquefaction rate. However, too much catalyst (i.e., in excess of 3%) will increase the residue content. The liquefaction efficiency is significantly improved by an increase in the liquid ratio. Experimental data suggest that both the decomposition of bagasse and the recondensation of the fragments are accelerated by an elevation of the reaction temperature.

The reaction time has little influence on the molecular weight of the liquefaction product.

Each liquefaction product comprises two major fractions having different molecular weights. The molecular weight distribution of each fraction is different when the reaction time increases.

The addition of liquefied bagasse can improve the adhesive shear strength of the cured epoxy resins.

The TGA curve of the cured resin polyblended with liquefied bagasse has two regions representing the two steps of the thermal degradation. The cured resin polyblended with liquefied bagasse presents a higher T_g than the pure epoxy resin cured under the same conditions. The variations of T_g and the degradation temperature indicate that the addition of liquefaction bagasse can improve the thermal stability of the epoxy resin.

References

1. Fierz-David, H. E. *Chem Ind Rev* 1925, 44, 942.
2. Miller, I. J.; Fellows, S. K. *Nature* 1981, 289, 398.
3. Appell, H. R.; Fu, Y. C.; Friedman, S. U.S. Bureau of Mines Investigation Report No. 7560; 1971.
4. Lin, L.; Yoshioka, M.; Yao, Y.; Shiraishi, N. *J Appl Polym Sci* 1994, 52, 1629.
5. Yao, Y.; Yoshioka, M.; Shiraishi, N. *Mokuzai Gakkaishi* 1995, 41, 659.
6. Shiraishi, N.; Onodera, S.; Ohtani, M.; Masumoto, T. T. *Mokuzai Gakkaishi* 1985, 31, 418.
7. Yao, Y.; Yoshioka, M.; Shiraishi, N. *Mokuzai Gakkaishi* 1994, 40, 176.
8. Kurimoto, Y.; Takeda, M.; Doi, S.; Tamura, Y.; Ono, H. *Bioresource Technol* 2001, 77, 33.
9. Kobayashi, M.; Tsukamoto, K.; Tomita, B. *Holzforchung* 2000, 54, 93.
10. Kobayashi, M.; Hatano, Y.; Tomita, B. *Holzforchung* 2001, 55, 667.
11. Lee, S.-H.; Teramoto, Y.; Shiraishi, N. *J Appl Polym Sci* 2002, 83, 1482.
12. Lee, S. H.; Yoshioka, M.; Shiraishi, N. *J Appl Polym Sci* 2000, 77, 2901.
13. Yamada, T.; Ono, H. *Bioresource Technol* 1999, 70, 61.
14. Feldman, D.; Banu, D.; Natansohn, A.; Wang, J. *J Appl Polym Sci* 1991, 42, 1537.
15. Kelley, S. S.; Glasser, W. G. *J Wood Chem Technol* 1988, 8, 341.
16. Hofmann, K. *J Wood Chem Technol* 1993, 13, 73.
17. Oliveira, W. D.; Glasser, W. G. *J Appl Polym Sci* 1989, 37, 3119.
18. Riddick, J. A.; Bunger, W. B.; Sakano, T. K. *Organic Solvents, Physical Properties and Methods of Purification*, 4th ed.; Wiley: New York, 1986.
19. Shirashi, N.; Shirakawa, K.; Kurimoto, Y. *Jpn. Pat. Appl.* 106128 (1992).
20. Mirbas, D. A. *Energy Conver Manage* 2000, 41, 633.
21. Yao, Y. Y.; Yoshioka, M.; Shiraishi, N. *Mokuzai Gakkaishi* 1993, 39, 930.
22. Peppel, W. J. *Ind Eng Chem* 1958, 50, 767.