

Measured kinetics of the acid-catalysed hydrolysis of sugar cane bagasse to produce xylose

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Abstract

Experimental trials of the water hydrolysis of bagasse to produce xylose, arabinose and glucose were conducted using a temperature-controlled microwave digester. The experimental variables were temperature, ratio of water mass to bagasse mass, type of bagasse material and reaction time. The pH of the liquid and concentration of dissolved xylose, arabinose and glucose were measured at the completion of each trial. Kinetic modelling of the global rates of formation of monosaccharide products was performed using schemes based on earlier researchers' models of acid hydrolysis using mineral acids. For the most plentiful product, xylose, the most accurate kinetic model of the global reactions was determined to be two parallel pathways for hydrolysis of xylan to xylose followed by a single pathway for xylose decomposition. The calculated activation energies of the reactions were within the range reported by other researchers for the hydrolysis of a range of lignocellulosic materials using mineral acids. © 2000 Elsevier Science B.V. All rights reserved.

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1. Introduction

Sugar cane bagasse constitute the fibrous residue of sugar cane after undergoing conventional milling. During a typical cane crushing season, about 50% of this fibre is needed to generate heat and power to run the sugar milling process [1], the remainder is stockpiled. The stockpiled bagasse is of low economic value and constitute an environmental problem to sugar mills and surrounding districts, especially if stockpiled for extended periods, due to the risk of spontaneous combustion occurring within the pile [2]. As a means of minimising this hazard, many mills resort to operating their furnaces in a highly inefficient manner so as to burn large portions of the excess bagasse. It has been

estimated [3,4] that bagasse consumption within sugar mill boilers could be reduced by up to 36% by ceasing such practices and adopting more energy efficient modes of operation. If a suitable use could be found for the excess bagasse there is, potentially, 2 million tonnes per annum of bagasse feedstock available in Australia alone [5].

As bagasse fibre is constituted, mainly, of lignocellulosic material, considerable research effort has been expended on investigating acid-catalysed hydrolysis to cleave the intrachain linkages in hemicellulose and cellulose chains contained in bagasse to produce commercial quantities of xylose, glucose and other sugars. Prior research on the hydrolysis of bagasse and similar materials has concentrated on the use of low concentration mineral acids [6–13]. In this paper research is presented on a simpler method of achieving the hydrolysis of bagasse hemicellulose by using hot water at

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elevated pressure. In this technique, water at elevated temperatures softens the lignin protective layer around the hemicellulose fibres and allows uronic, glucuronic and acetic acids to be released from the polymeric backbone of hemicellulose and lignin [6]. These acids act as a catalyst for the hydrolysis of the hemicellulose to form polysaccharides and monosaccharides of, mainly, xylose, arabinose and glucose. With sustained hydrolysis, the sugars are degraded to such decomposition products as furfural, hydroxymethylfurfural and furan resins. As xylan is the major constituent of hemicellulose, xylose is the major potential product of this process and the focus of this study.

In the research presented here, the reaction conditions tested were temperature (80–240°C), ratio of bagasse mass to water mass (1:5–1:30), type of bagasse material (i.e. bagasse, bagacillo, pith or rind), and reaction time (10–4000 min). The pH of the liquid and concentration of dissolved xylose, arabinose and glucose were measured at the completion of each trial. Kinetic modelling of the global rates of formation of monosaccharide products, based on earlier researchers' models of acid hydrolysis using mineral acids, is presented.

2. Experimental

2.1. Characterisation of materials tested

Four bagasse fractions were used in the study: (1) mill-run bagasse, (2) bagacillo, (3) pith and (4) rind. The latter two fractions were available from a 'cane separator' [14], a device for separating whole cane stalks into pith, epidermis and rind fractions. All four feed samples were taken from a single mill, Victoria mill. The terms 'mill-run bagasse' and bagasse are interchangeable and refer to samples of sugar cane fibre collected from the last tandem roller in the milling

train. Bagacillo constitutes the finer size fraction of bagasse that is used as a filter aid in the refining process. It was separated from the bagasse by a cross-flow of air blown through a stream of falling bagasse.

The standard TAPPI method (method T 223 cm-84) [15] was applied to determine the pentosan (combined xylan and arabinan) content of each of the bagasse fractions (see Table 1). The repeatability within a laboratory was reported in the method as 6.6% and the analytical error was calculated using this figure. Pith had the highest pentosan content, even allowing for sampling and analytical error. Bagacillo and bagasse had, as expected, similar pentosan content. The rind fraction contained the least potential xylose. All pentosan contents of the various bagasse fractions studied in this work fell within the range 192–323 mg/g that have been reported for bagasse [16].

The ratio of arabinan to xylan in bagasse was estimated to be 1.2:10. This value was based on an average of previously reported figures [8,12,17–19] and the observation that the ratio of arabinose to xylose formed in hydrolysis experiments conducted at relatively low temperatures (when degradation of the monosaccharides is negligible) approached this value. Similarly, the ratio of glucuronic acid (which, when hydrolysed, forms glucose) to xylan was estimated to be 1:10.

2.2. Hydrolysis experiments

All bagasse materials tested were dried at 105°C and stored in a desiccator. For hydrolysis tests, a sample of bagasse (0.750–1.000 g) was placed in an 86 ml Teflon reaction vial. The desired volume of water (5–15 ml) was pipetted into the vial and the vial then sealed. The Teflon reaction vial was mounted in a Milestone MLS 1200 microwave oven. The vial contained a temperature sensor connected to the microwave control unit to ensure rapid heating and accurate temperature control. The heating time to the desired temperature was

Table 1
Chemical content of dried bagasse fractions

Bagasse fraction	Pentosan (mg/g) material (dry basis)	Analytical error within laboratory (mg/g)	γ
Pith	317	±21	0.523
Bagacillo	277	±18	0.677
Bagasse	268	±18	0.576
Rind	225	±15	0.417

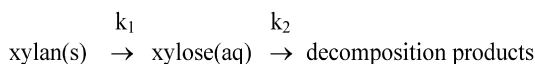
of the order of 2–3 min. At the end of the reaction time period the sample was cooled promptly using a water cooled bath. The product was filtered and washed with hot water until the filtrate volume had reached 50 ml.

The bagasse fraction residue filter cake was washed, dried and weighed. As the quantity of dry feed bagasse was measured before hydrolysis, the percentage of bagasse digested during the test could be determined. Samples of the filtrate were analysed for xylose, arabinose and glucose by HPLC. The analysis conditions used to measure xylose, arabinose and glucose concentrations were column: $300 \times 6.5 \text{ mm}^2$, packing: interaction CHO-620, mobile phase: $0.05 \text{ mol/dm}^3 \text{ Na}_2\text{Ca (EDTA)}$, flow rate: 0.5 ml/min , pre-column 1 (Keystone Scientific): C-18 guard column ($11 \times 6 \text{ mm}^2$), pre-column 2: interaction CHO-620 guard column ($23 \times 6 \text{ mm}^2$), temperature: 90°C , injection volume: $20 \mu\text{l}$, and run time: 22 m.

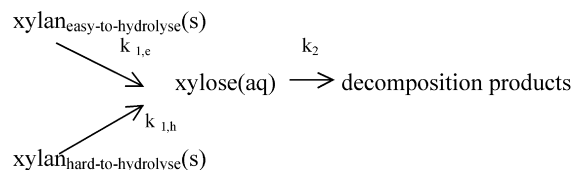
3. Kinetic models

3.1. Xylose formation

A variety of models for hemicellulose acid hydrolysis are postulated in the literature [6]. The simplest model for xylan hydrolysis involves a series of irreversible reactions from solid xylan to aqueous xylose and then onto decomposition products (Scheme 1). This scheme has often been considered deficient, as the decomposition of xylan to xylose cannot be considered a single step process [20]. Rather, the xylan chain is first decomposed to xylo-oligosaccharides, then the decomposition proceeds to yield xylose monomer [21]. The xylose decomposes further to furfural and then onto furan resins and other products. For the purpose of practical calculation, the decomposition of xylan has often been modelled as the sum of two parallel first-order reactions, namely a fast hydrolysis reaction and a slow hydrolysis reaction (Scheme 2). Consequently, the xylan has been categorised into an ‘easy-to-hydrolyse’ fraction and



Scheme 1.



Scheme 2.

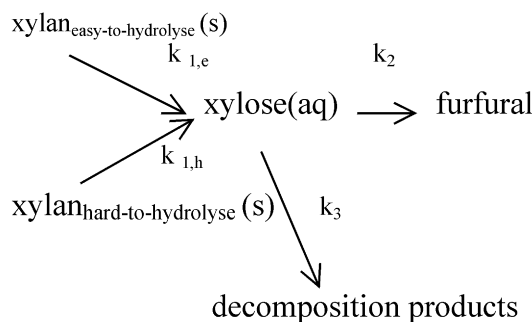
a ‘hard-to-hydrolyse’ fraction [6,22,23]. It must be noted that these fractions have been defined for convenience of rate calculations and, in previous work, has not been linked to any physical or chemical characteristics of the raw material.

Scheme 2 has been further refined to take account of the possibility of multiple decomposition products that may form from xylose [24,25]. Consequently, a different mechanism (Scheme 3) has been proposed [6]. In this scheme, furfural is the major decomposition product with known activation energy for the decomposition process (140.5 kJ/mol [26]).

In all schemes, the reaction rate has been assumed to be first-order with respect to the reactants in each reaction step. Under conditions of isothermal reaction, the concentration of xylose present in the aqueous solution for Schemes 1–3 are, respectively,

$$C_x^L = C_{x0}^s \left(\frac{k_1}{\phi k_2 - k_1} \right) (e^{-k_1 t} - e^{-\phi k_2 t}) \quad (1)$$

$$\begin{aligned} C_x^L = C_{x0}^s & \left(\left(\frac{\gamma k_{1,e}}{\phi k_2 - k_{1,e}} \right) (e^{-k_{1,e} t} - e^{-\phi k_2 t}) \right. \\ & \left. + \left(\frac{(1-\gamma) k_{1,h}}{\phi k_2 - k_{1,h}} \right) (e^{-k_{1,h} t} - e^{-\phi k_2 t}) \right) \quad (2) \end{aligned}$$



Scheme 3.

$$C_x^L = C_{x0}^s \left(\left(\frac{\gamma k_{1,e}}{\phi(k_2 + k_3) - k_{1,e}} \right) \times (e^{-k_{1,e}t} - e^{-\phi(k_2 + k_3)t}) + \left(\frac{(1-\gamma)k_{1,h}}{\phi(k_2 + k_3) - k_{1,h}} \right) \times (e^{-k_{1,h}t} - e^{-\phi(k_2 + k_3)t}) \right) \quad (3)$$

where C_x^L is the concentration of xylose in liquid (g/g bagasse), C_{x0}^s the equivalent xylose concentration if all xylan were converted to xylose (g/g bagasse), k_i the reaction rate constant for reaction step i (s^{-1}). The rate constant is assumed to obey an Arrhenius relationship with activation energy E_i (J/mol) and pre-exponential constant A_i (s^{-1}), t the reaction time (s), γ the fraction of xylan that is easy-to-hydrolyse, and ϕ the ratio of solid bagasse material to liquid (g/g).

In analysing the xylose yields from the experimental trials, these three equations were fit to the results to determine which scheme best agreed with the experimental data. The unknown adjusted parameters were the activation energies and pre-exponential factors.

3.2. Arabinose and glucose

Kinetic models of the formation of arabinose and glucose by hemicellulose hydrolysis have not been

reported in the literature. It was decided to test a model of the formation of these products using consecutive first-order reactions in series identical to Scheme 1.

4. Results and discussion

4.1. Determination of the easy-to-hydrolyse and hard-to-hydrolyse xylan fractions

As noted in the previous section, the modelling of the xylose formation by hemicellulose hydrolysis has often required the determination of the easy-to-hydrolyse and hard-to-hydrolyse fraction of the xylan. To determine these fractions, bagasse samples were hydrolysed with 4% sulphuric acid at 90°C in 1 l reagent bottles placed in a water bath at 90°C. Samples of hydrolysate were taken at regular intervals and analysed for xylose. Under these mild conditions, degradation of the xylose is minimised as the hydrolysis of xylan to xylose occurs. Fig. 1 shows a plot of $(1 - C_x^L/C_{x0}^s)$ versus time for the different bagasse materials tested. It can be seen that the concentration of xylose increases relatively rapidly over the first 300–600 min and follows a trajectory that would be expected for a first-order reaction. This period has

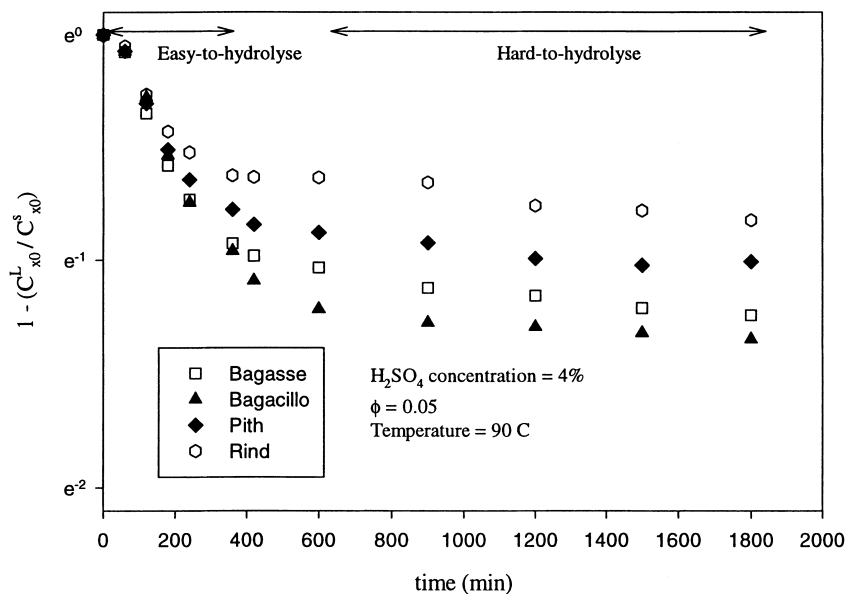


Fig. 1. Determination of hard-to-hydrolyse xylan fraction in bagasse material.

been interpreted as representing the hydrolysis of the easy-to-hydrolyse fraction of the xylan. The hydrolysis then proceeded at a slower but measurable rate. This period has been interpreted as representing the hydrolysis of the hard-to-hydrolyse fraction of the xylan. The fraction of easy-to-hydrolyse material (γ) was determined by extrapolating the data points, for times greater than 400 min, back to time = 0. From this analysis, the value of γ was determined (see Table 1). These results indicated a strong variation in the xylan degradation rates existed between the different types of bagasse materials with the pith and rind material containing the higher amounts of hard-to-hydrolyse xylan. A possible explanation for this could be based on the way in which these materials were prepared. Whereas the bagasse and bagacillo materials underwent a significant amount of crushing and grinding in the sugar milling process, the pith and rind materials were sourced from the cane separator which involved much less disruption of the materials' constituent cells. This would tend to reduce the area available for surface reaction and inhibit the diffusion of reactants and products within the materials.

4.2. Xylose kinetics results

A total of 131 separate hydrolysis experiments were conducted with the following reaction conditions: temperature (80–240°C), ratio of bagasse mass to water mass (1:5–1:30), type of bagasse material (i.e. bagasse, bagacillo, pith or rind), and reaction time (10–4000 min). During all experiments the pH of the hydrolysis solution was found to have a pH ranging between 4 and 2.4. It was assumed that this level of variation in acid concentration would have a

negligible effect on the rate of the catalytic process. A Marquardt–Levenberg curve-fitting routine available in Sigmaplot Version 2.01 was used to fit Eqs. (1)–(3) to the experimental data using the activation energies and pre-exponential constants as adjustable parameters. The predicted results from the curve-fitting routine were compared with the experimental results and analysed to determine the standard error and regression coefficient by ANOVA. The predicted parameters, regression coefficients and standard error for each scheme is given in Tables 2–4. The tables clearly show the predicted xylose yields for the three different models give very similar levels of accuracy, the variation between the predictions of the different models are negligible. Scheme 2 gives slightly low standard error, and it is interesting to note that in this model the rate of hydrolysis of the hard-to-hydrolyse xylan is zero, indicating that some portion of the xylan is inert to the hydrolysis process. The activation energy for xylan hydrolysis to xylose determined for all bagasse materials (99.3–130 kJ/mol) were in the same range as has been reported for a range of lignocellulosic materials (53–142 kJ/mol) [8,13,20,27,28].

Fig. 2 shows typical results for the predicted and experimental data for bagasse hydrolysis to produce xylose as a function of time. There is some scatter in the experimental data that is ascribed to the variation in bagasse properties between tests and analytical error. The plots show a clear increase in xylose concentration as the xylan degrades, reaching a maximum, then decreasing when the xylose degradation rates become significant. At the higher temperature (200°C), the rates of reaction are increased and the maximum xylose concentration is reached after less time. At the higher value of ϕ (0.2), as the xylose

Table 2
Kinetic model solutions for hydrolysis of xylan in different bagasse feeds (Scheme 1)

Feed	Derived data			
	Bagacillo	Bagasse	Pith	Rind
A_1 (s^{-1})	0.0386×10^9	0.164×10^9	0.955×10^9	44.92×10^9
E_1 (kJ/mol)	99.4	105.0	111.8	126.3
A_2 (s^{-1})	6.44×10^{10}	2.76×10^{11}	2.43×10^{11}	1.42×10^{13}
E_2 (kJ/mol)	111.9	119.3	118.9	134.7
Correlation coefficient, R	0.8763	0.8214	0.9068	0.9291
Standard error in xylose yield (mg/g)	13.1	16.7	17.1	11.0

Table 3

Kinetic model solutions for hydrolysis of xylan in different bagasse feeds (Scheme 2)

Feed	Derived data			
	Bagacillo	Bagasse	Pith	Rind
$A_{1,e}$ (s^{-1})	0.0674×10^9	0.128×10^9	1.308×10^9	14.46×10^9
$E_{1,e}$ (kJ/mol)	100.2	102.0	110.6	117.2
$A_{1,h}$ (s^{-1})	0	0	0	0
$E_{1,h}$ (kJ/mol)	—	—	—	—
A_2 (s^{-1})	1.59×10^{11}	2.12×10^{11}	2.04×10^{11}	3.26×10^{11}
E_2 (kJ/mol)	116.4	120.3	120.7	123.6
Correlation coefficient, R	0.8825	0.8231	0.9083	0.9059
Standard error in xylose yield (mg/g)	12.8	16.6	16.9	12.6

Table 4

Kinetic model solutions for hydrolysis of xylan in different bagasse feeds (Scheme 3)

Feed	Derived data			
	Bagacillo	Bagasse	Pith	Rind
$A_{1,e}$ (s^{-1})	37.9×10^6	158.2×10^6	1.126×10^9	126.8×10^{11}
$E_{1,e}$ (kJ/mol)	99.3	104.8	112.5	130.3
$A_{1,h}$ (s^{-1})	37.9×10^6	158.2×10^6	1.126×10^9	126.8×10^{11}
$E_{1,h}$ (kJ/mol)	99.3	104.8	112.5	130.3
A_2 (s^{-1})	3.09×10^{10}	3.92×10^{10}	5.22×10^{13}	6.46×10^{13}
E_2 (kJ/mol) (all the same)	140.5	140.5	140.5	140.5
A_3 (s^{-1})	6.36×10^{10}	2.59×10^{11}	1.06×10^4	3.63×10^{-12}
E_3 (kJ/mol)	111.9	119.1	62.1	10.0
Correlation coefficient, R	0.8763	0.8213	0.9164	0.9259
Standard error in xylose yield (mg/g)	13.4	16.9	16.5	11.5

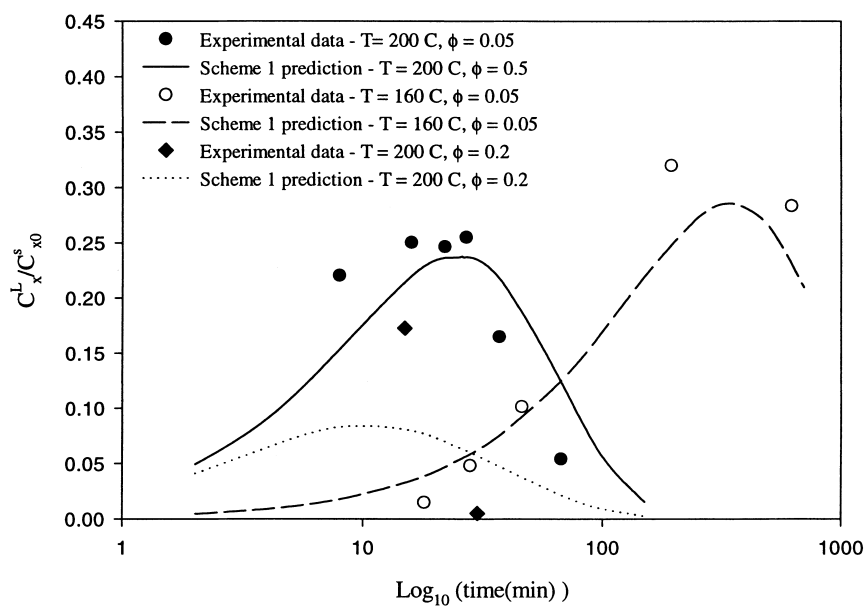


Fig. 2. Xylose yields by bagasse hydrolysis.

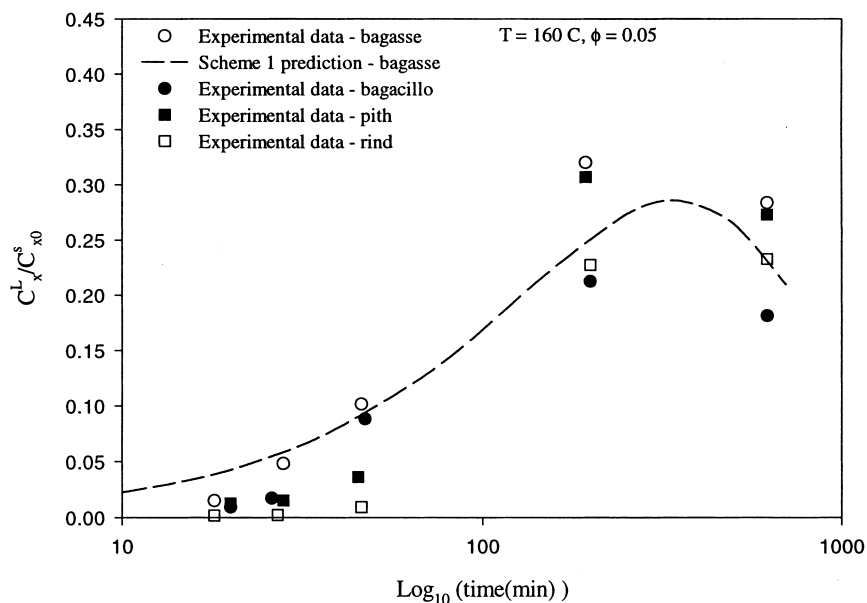


Fig. 3. Xylose yields for different bagasse materials.

produced is concentrated in the aqueous concentration the rate of xylose degradation is increased thus decreasing the potential maximum yield of xylose.

Fig. 3 compares the xylose yields obtained from the different bagasse materials. There is some variation in the yields with no clear correlation between the proportion of easy-to-hydrolyse material and the xylose yield. The curves appear similar with the time for maximum yield being the same for all materials. There is no evidence that diffusion through the bagasse fibres is affecting the rate of reaction as the finer materials (bagacillo and pith) show no improvement in yield over the coarser materials bagasse and rind.

In all tests performed, the xylose yield did not exceed 30% of the potential yield. However, weight loss measurements on the bagasse show the mass of bagasse consumed during hydrolysis exceeded considerably (up to 2.5 times) the amount of xylose formed (see Fig. 4). As the lignins and cellulose in bagasse are known to be virtually insoluble under the reaction conditions, it is hypothesised that the mass loss is mainly due to the partial hydrolysis of the hemicellulose to short chain polymers that are soluble

in the aqueous phase and where further hydrolysis occurs.

4.3. Arabinose and glucose kinetics results

Tables 5 and 6 present the predicted activation energies and pre-exponential constants for the production of glucose and arabinose from the bagasse materials. As with the xylose results, there was some scatter in the experimental data. The tables show the standard predicted error is given for the kinetic models. For the glucose results, significant experimental error was found due to the presence of residual sucrose within the bagasse material, especially in the cane separator samples. This sucrose was inverted during hydrolysis trials resulting in excessively high glucose yields. Thus, only results for bagasse and bagacillo are given in Table 6. The percentage yield of arabinose was found to be significantly higher than the xylose yield, with up to 60% of the theoretical arabinose content being produced in some trials. The higher yield may be due to the structure of hemicellulose polymer, which contains xylan as the fibre backbone with arabinose monomers attached to side chains [17], and

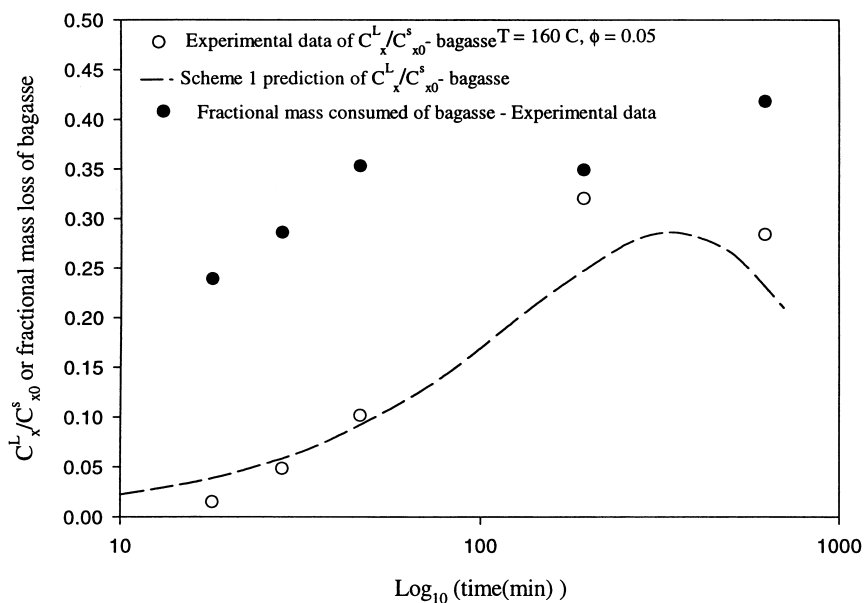


Fig. 4. Comparison of xylose yields with mass consumption of bagasse.

Table 5

Kinetic model solutions for the formation of arabinose in different bagasse feeds (Scheme 1)

Feed	Derived data			
	Bagacillo	Bagasse	Pith	Rind
A_1 (s^{-1})	7.28×10^6	10.1×10^6	25.6×10^6	383×10^6
E_1 (kJ/mol)	89.0	91.4	98.1	114.9
A_2 (s^{-1})	3.20×10^{10}	0.380×10^{10}	1.14×10^{10}	0.00600×10^{10}
E_2 (kJ/mol)	111.4	103.8	107.8	89.2
Correlation coefficient, R	0.8894	0.8242	0.8050	0.6672
Standard error in arabinose yield (mg/g)	3.2	2.5	2.9	1.7

hence the arabinose is more easily cleaved from the hemicellulose.

Table 6

Kinetic model solutions for the formation of glucose in different bagasse feeds (Scheme 1)

Feed	Bagacillo	Bagasse
A_1 (s^{-1})	44.8×10^6	18.0×10^{12}
E_1 (kJ/mol)	104.9	123.3
A_2 (s^{-1})	21.8×10^{12}	0.190×10^9
E_2 (kJ/mol)	141.7	127.8
Correlation coefficient, R	0.7637	0.3952
Standard error in glucose yield (mg/g)	4.2	4.6

5. Conclusions

Three kinetic models of xylose production by the water hydrolysis of bagasse have been tested. The most accurate model was given by Scheme 2, however, the other schemes gave almost equally accurate predictions. The predicted value of activation energy for the xylan degradation in the bagasse materials were of the same order as previously reported for other lignocellulosic materials. The four types of bagasse mate-

rial tested (mill-run bagasse, bagacillo, rind and pith) contained different quantities of xylan material and yielded significantly different amounts of xylose product when subjected to the same reaction conditions. However, the time required for maximum xylose yield was similar for all materials. During hydrolysis, the pH of the mixture rapidly decreased to ~3–4 before stabilising in this region. Measurement of the weight loss of bagasse during hydrolysis indicates the hemicellulose material is partially hydrolysed during the reaction to form short chain polymers which then undergo further degradation in the liquid phase.

A simple consecutive series reaction scheme was used to model the production of arabinose and glucose from the bagasse materials. The models gave similar levels of accuracy as the xylose results. The percentage yields of these products were significantly higher than that of xylose.

Acknowledgements

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