PYROLYSIS KINETICS OF RICE HUSK

S. Tia*, S. Suwanayuen, N. Khamdee

Combustion Research Laboratory
Department of Chemical Engineering
King Mongkut's Institute of Technology Thonburi
Bangkok 10140, Thailand

ABSTRACT

Pyrolysis kinetics of rice husk were estimated by means of a non-isothermal thermogravimetric technique. The results obtained suggested that the pyrolysis experiment which was performed with fine particles (below 150 microns) in a small sample mass (below 10 mg) and at a low heating rate was controlled by chemical kinetics. The pyrolysis of rice husk was well predicted by a multireaction model based on the main biomass components (cellulose, hemicellulose, lignin) model. The distributed activation energy model, which is widely used in the literature, showed fairly good agreement with our experiment. With the assumption of no temperature gradient inside the husk, the former model, with which the transient heat transfer equation was incorporated, showed satisfactory weight loss prediction of a single rice husk particle pyrolyzed inside a constant temperature furnace.

INTRODUCTION

Thailand, the world's major rice exporter, produces about 5 Mtons of rice husk each year[1]. The utilization of this residue is mainly as fuel in the combustion process of the rice mill. The pyrolysis/gasification of rice husk has also been considered for obtaining gas fuel for power generation using a modified diesel engine[2]. In addition, the ash obtained from these conversion processes with little carbon content can be used as a source of silicon for various products such as insulating/refractory material, polishing compounds and silicone chloride production[3,4]. Since the volatile content of rice husk can contribute about 50% or more of it's total heating value, the study of pyrolysis, which is the first step in any thermal conversion process, is therefore important. As reviewed in the literature[5], the pyrolysis of biomass is effected by its composition, reaction temperature, heating rate, paricle size, and ambient gas. For kinetic control regime, it was reported that the devolatilization of biomass in an inert atmosphere was well predicted by the multireaction models based on distributed activation energy[6], main biomass components[7], and volatile products[8]. The first two models can predict only total weight loss of biomass. The last, which needs more kinetic parameters, can predict both total weight loss and product distribution evolved during pyrolysis.

This paper reports on the study of rice hush pyrolysis using the non-isothermal thermogravimetric technique. The effect of particle size and sample mass on the weight loss history were also studied. The models reported in the literature were used for weight loss prediction and compared with the experimental data from both thermogravimetric Analyzer (TGA) and captive sample technique.

THEORY

a.) Distributed Activation Energy (DAE) Model

This model treats the decomposition of solid fuels as a large number of independent, parallet first-order reactions and the activation energy of all reactions can be presented by the continuous Guassian distribution. Assuming the same frequency factor in the Arrhenius rate expression, the non-isothermal weight loss of a solid particle at constant heating rate and zero hold-up time can be approximated as[9]:

$$1-x = \frac{1}{\sqrt{\pi}}\int_{-\infty}^{\infty}\exp(-y^2)dy \qquad (3)$$

$$= \frac{1}{2}\,erfc\,(y_c) \qquad (4)$$

$$= \frac{1}{2}\,[1-erf(y_c)] \qquad (5)$$

and

$$y = (z-z_o)/g \;,\; y_c = (z_c-z_o)/g \qquad (6)$$

where x = fraction of weight loss $[(w_o-w)/(w_o-w_f)]$, w_o = initial weight of sample, w = sample weight at time t, w_f = sample weight at t approaching infinity, $g = \sigma\sqrt{2}/RT$, $z = E/RT$, $z_o = E_o/RT$, E = activation energy, R = universal gas constant, T = absolute temperature, E_o = mean activation energy, and σ = standard deviation of E's in all reactions from E_o. The value of z_c is estimated from[6]:

$$\frac{A}{z_c}\exp(z_c) = 0.5 \qquad (7)$$

where $A = k_o/RT$ and k_o = frequency factor. This approximation gave an error below 5% for A = 10^5-10^{20}, z_o = 10-100, and g = 2-20[9].

This model, with k_o, E_o, σ, and $(w_o - w_f)$ as the kinetic parameter, is the most commonly used in coal pyrolysis[10].

b.) Model based on main biomass components

This model, which was proposed by Koufopanos et al.[7], considered the pyrolysis rate of small biomass particles as being the sum of the rate of main biomass components. The rate contributed by each component is proportional to its composition in the virgin biomass. The interactions among the components were assumed negligible. With these assumptions, the components in biomass are[7]:

Biomass -----> a[Cellulose] + b[Lignin] + c[Hemicellulose] (8)

where a, b, and c are the weight fraction of corresponding components in the virgin biomass. The pyrolysis reaction of each component is[7]:

Virgin Material --1--> Intermediate --2--> Gases + Tars

Intermediate --3--> Char

The first reaction, of which the assumed reaction order is zero, describes the changes in the chemical structure of the biomass at low temperature and does not influence the observed weight loss. This unstable product continues to decompose to volatile gas (gases and tars) and char as two competitive reactions. The pyrolysis rate of these reactions are:

$$\frac{dB}{dt} = -k_1 \exp\left(\frac{-E_1}{RT}\right) B^{n1} \qquad (9)$$

$$\frac{d(BI)}{dt} = k_1 \exp\left(\frac{-E_1}{RT}\right) B^{n1} - k_2 \exp\left(\frac{-E_2}{RT}\right) (BI)^{n2} - k_3 \exp\left(\frac{-E_3}{RT}\right) (BI)^{n3} \qquad (10)$$

$$\frac{dC}{dt} = k_3 \exp\left(\frac{-E_3}{RT}\right) (BI)^{n3} \qquad (11)$$

Where B, BI, and C are the weight faction of virgin material, intermediate, and char respectively, k_i = pre-exponential factor for reaction i (i = 1, 2, and 3), E_i = activation energy for reaction i, and n_i = order of reaction i. The initial boundary conditions are:

$$B = 1, \quad BI = C = 0 \qquad (12)$$

and the weight retained by each component (w_j, j = cellulose, lignin, and hemicellulose) is

$$w_j = B + BI + C \qquad (13)$$

The kinetic parameters for pysolysis reaction of each component were proposed by Koufopanos et al.[7] as presented in Table 1.

EXPERIMENT

a.) Sample Preparation

Rice husk sample, of which the properties are shown in Table 2, was ground and sieved to different fractions. After overnight drying at 378 K, the samples were kept in a desiccator.

b.) Experimental procedure

The experiments were carried out in a Thermogravimetric Analyzer (TGA, model TGS-2,

Table 1 Kinetic parameters for pyrolysis reaction of main biomass constituent.

Reaction		Cellulose	Hemicellulose	Lignin
1	n_1	0	0	0
	k_1	2.2×10^{14}	3.3×10^{6}	3.3×10^{12}
	E_1	167.5	72.4	147.7
2	n_2	1.5	1.5	1.5
	k_2	9.4×10^{15}	1.1×10^{14}	8.6×10^{8}
	E_2	216.6	174.4	137.1
3	n_3	1.5	1.5	1.5
	k_3	3.1×10^{13}	2.5×10^{13}	4.4×10^{7}
	E_3	196	172	122.1

Note: k_i and E_i are in $s^{-1}.g^{-1}$ and $kJ.mol^{-1}$, respectively.

Perkin-Elmer) connected with a Data Station under non-isothermal mode at various sample masses (2-7 mg), particle sizes (-90, 90-105, and 106-150 μm), and heating rates (5-50 K. min^{-1}). Nitrogen (99.99% purity at 200 $ml.min^{-1}$) was used as the purge gas. The final temperature of the heating furnace was 850-950 K.

In addition, the isothermal experiments for a whole husk in a tube furnace under nitrogen atmosphere using captive sample technique were also carried out. The inside diameter of the furnace is 4 cm. and its length is 34 cm. The temperature inside the furnace was measured by using a K-type thermocouple connected with a PID controller. After the furnace was preheated to the desired temperature the whole husk sample, which was fixed on the end of a ceramic needle, was one at a time quickly inserted into the funance. The percent weight retained of the sample at various residence times were recorded. The data obtained was then compared with the predicted result obtained from the theory.

RESULTS AND DISCUSSION

TG Curve

The typical TG curve obtained from rice husk pyrolysis which is the plot of sample weight as a function of temperature is presented in Fig. 1, as well as its differential (DTG curve). The sample shows the beginning of weight loss due to thermal decomposition at around 473 K. The ultimate yield of volatles are found to be about 65% by weight of dry sample at about 900 K (Fig. 1). These values are slightly higher than that obtained from proximate analysis (Table 2). This behavior, however, has been reported in the literature[12], and in this case may be due to the use of relatively small smaple mass in the

Table 2 Properties of rice husk sample[11].

Moisture content, %			10.21
Sphericity			0.24
Equivalent diameter, cm			0.214
Apparent density, $kg.m^{-3}$			847
Lower heating value, $kJ.kg^{-1}$			12,671
Proximate analysis (moisture-free basis, %)			
Volatile matter			60.90
Fixed carbon			23.00
Ash			16.10
Ultimate Analysis (moisture-free basis, %)			
Carbon	36.41	Hydrogen	4.90
Nitrogen	0.21	Oxygen	42.38
Ash	16.10		
Main components (dried ash-free basis, %)			
Cellulose	55.70	Hemicellulose	20.60
Lignin	21.10	Others	2.60
Ash composition (%)			
SiO_2	76.50	CaO	0.09
MgO	0.44	K_2O	4.80
Na_2O	0.07	P_2O_5	0.96
SO_3	0.54	Fe_2O_3	0.05
MnO_2	0.22	Others	16.33

experiment compared to proximate analysis. This will reduce the opportunity for secondary cracking with the neighboring particles[12]. A single peak of DTG curve, which is sensitive to the temperature change, is also observed. It can therefore be concluded that under this experimental condition the pyrolysis was controlled by chemical reaction.

However, when the whole husk was used as sample, the TG curve shifts to the right with the same ultimate yield of volatiles as the ground sample, and the significant change in slope at about 550 K for the DTG curve was also observed (Fig. 2). This may be due to the heat-up effect of the relatively large size of the whole husk sample, and thus the sample temperature is lower than TGA's furnace temperature. Once the thermal dissociation temperature was reached, the pyrolysis was also controlled by chemical reaction with little effect of mass transfer, as observed from the DTG curve which shows small decrease in sharp peak (Fig. 2). This behavior corresponds to those reported in the literature[7] for biomass with a particle size larger than 1 mm.

The TG curves for different sample mass having the same particle size (90-106 micron) are shown in Fig. 3. It was found that the 1-7 mg. sample size showed almost the same TG curves which confirmed the minimum effect of transport processes(heat and mass transfer) across the sample layer. Using 2 mg. sample mass, the pyrolysis was controlled by chemical reaction for particle size below 150 micron (Fig. 4). The pyrolysis of whole rice husk as discussed above is also shown for comparison. Figure 5 presents the effect of heating rate on rice husk pyrolysis. The decomposition tempra-ture increases as the heating rate increases but, within the experimental conditions of 5-50 K. min-1 heating rate, the asymptotic yield of residual char is the same. This can be attributed to the time for heating at a slow heating rate being longer than the pyrolysis time, and thus the reaction is complete at all pyrolysis tem-peratures. When the heating rate was increased the time required to achieve a given weight loss at any temperature decreased, and hence gave higher weight retention.

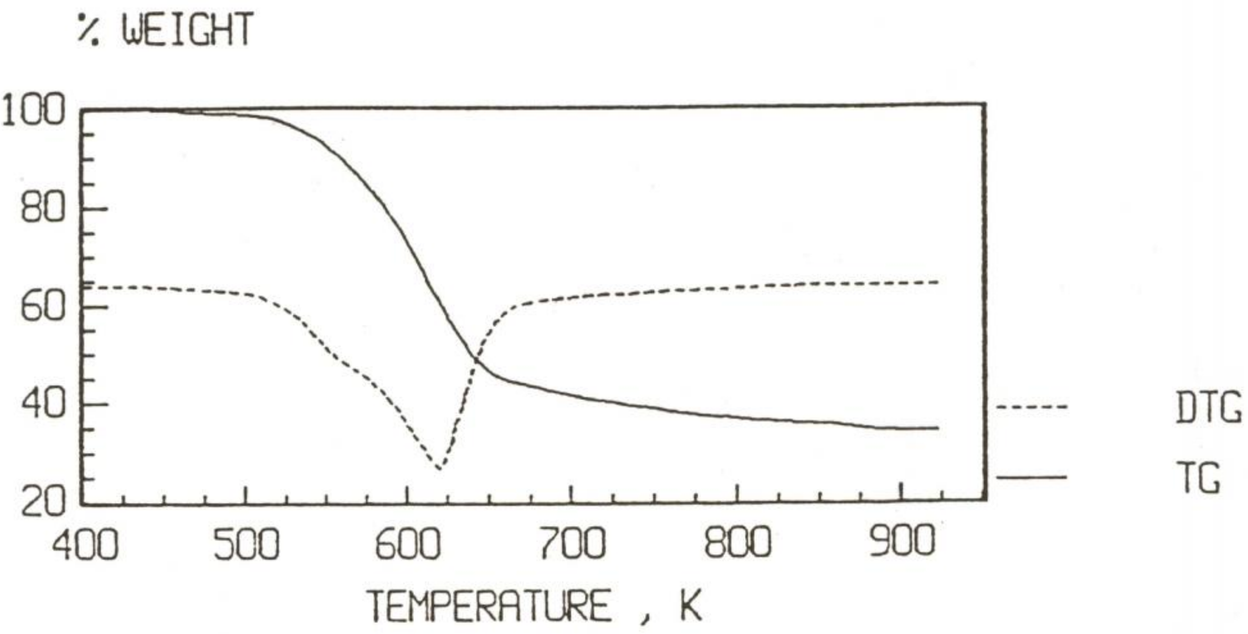


Figure 1 TG and DTG curves of rice husk pyrolysis. Condition: sample mass = 2 mg, heating rate = 5 $K.min^{-1}$, and particle size = 90-106 micron.

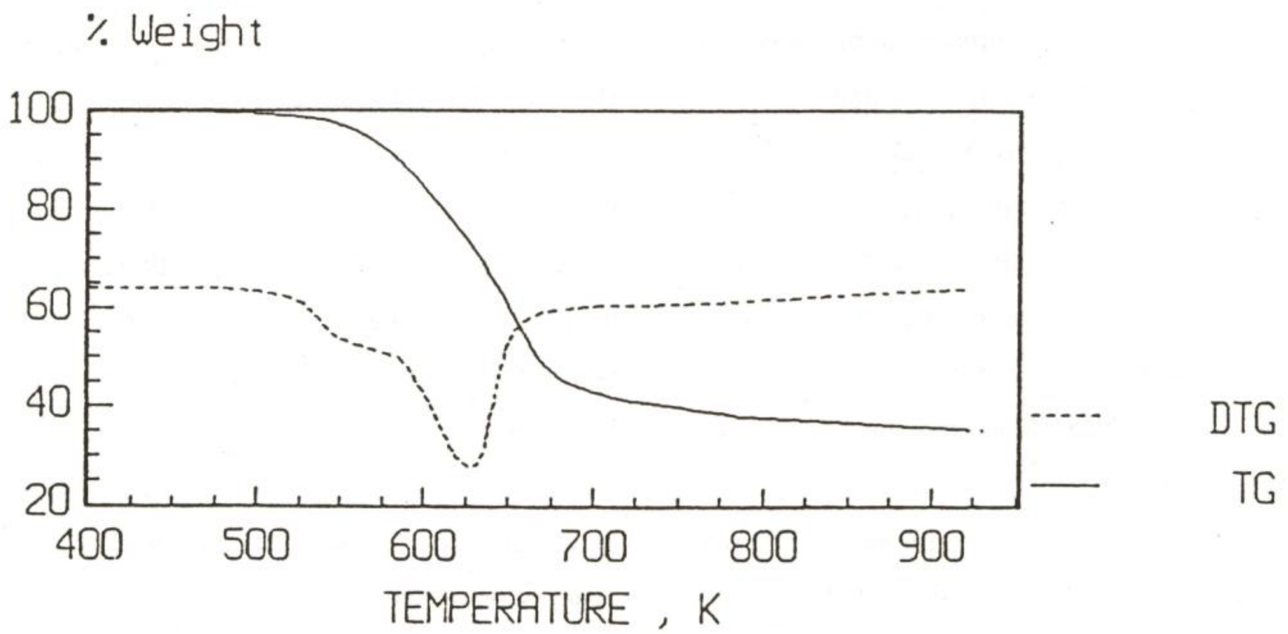


Figure 2 TG and DTG curves of whole rice husk pyrolysis.
Condition: heating rate = 5 $K.min^{-1}$.

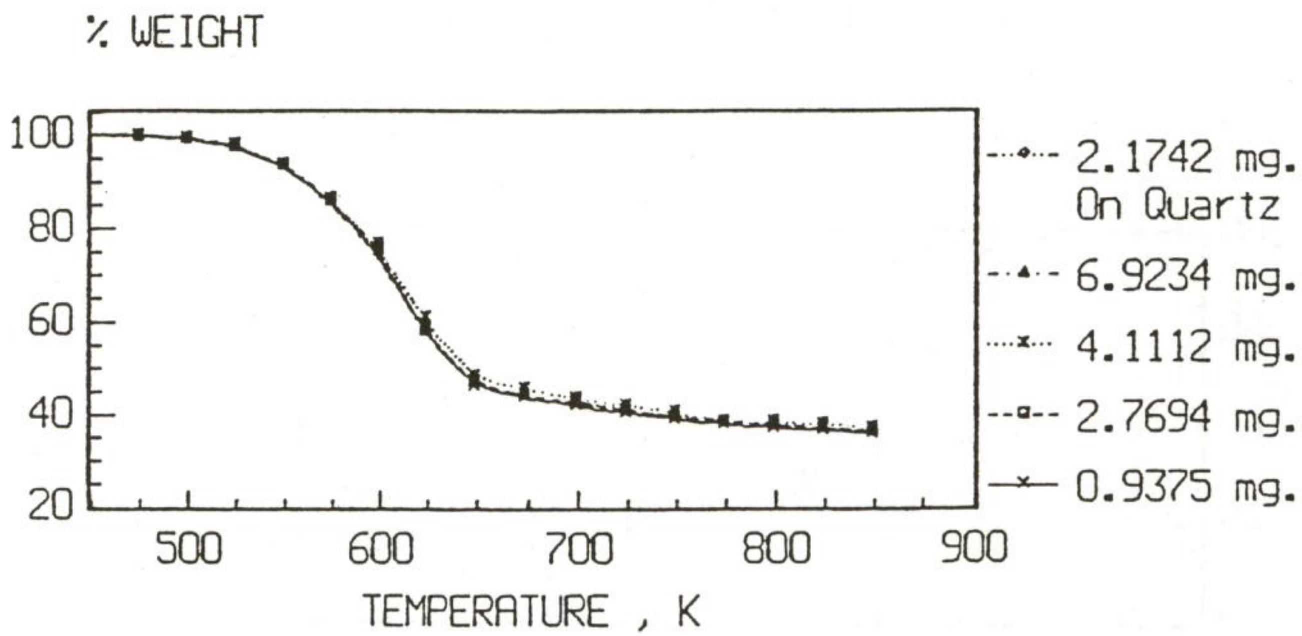


Figure 3 Effect of sample mass on rice husk pysolysis.
Condition: heating rate = 5 $K.min^{-1}$ and particle size = 90-106 micron.

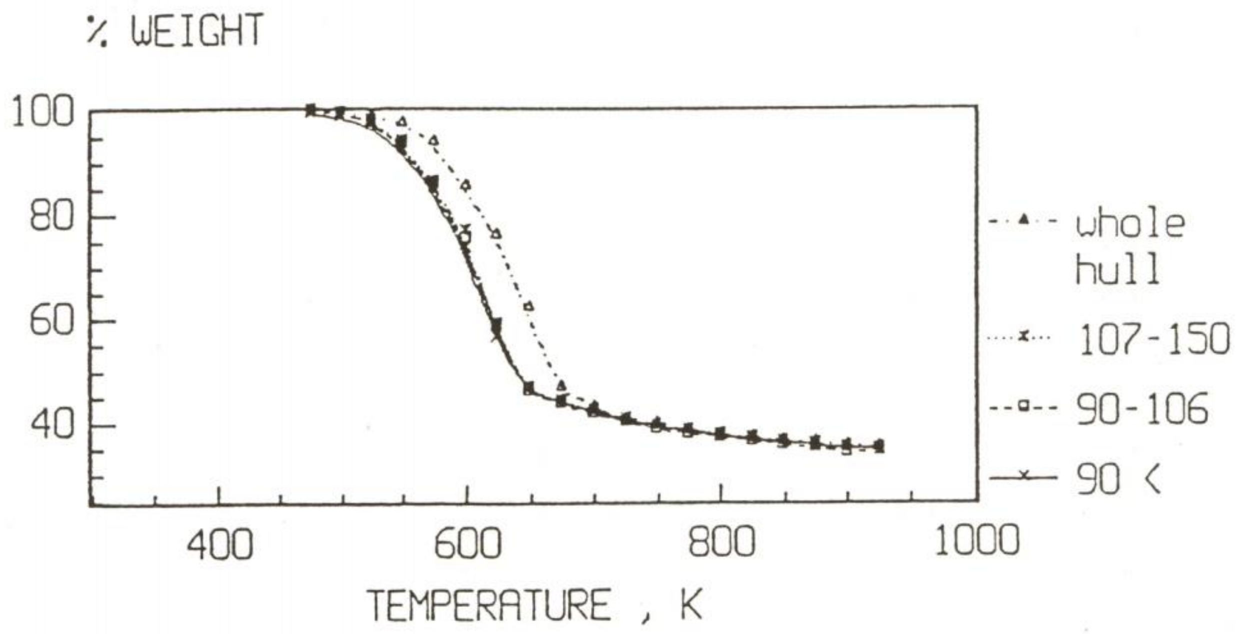


Figure 4 Effect of particle size on rice husk pyrolysis.
Condition: sample mass = 2 mg and heating rate = 5 K.min^{-1}.

Estimation of Kinetic Parameters

a.) Distributed Activation Energy Model

The set of equations given for the DAE model have been used to evaluate the kinetic parameters from non-isothermal TGA data. The frequency factor, k_o, which has been calculated from transition state theory, was fixed at 1.67×10^{13} s^{-1}[10], and the ultimate yield, (w_o-w_f), can be taken from experimental date (66.84% of dried rice husk). The number of parameters needed to

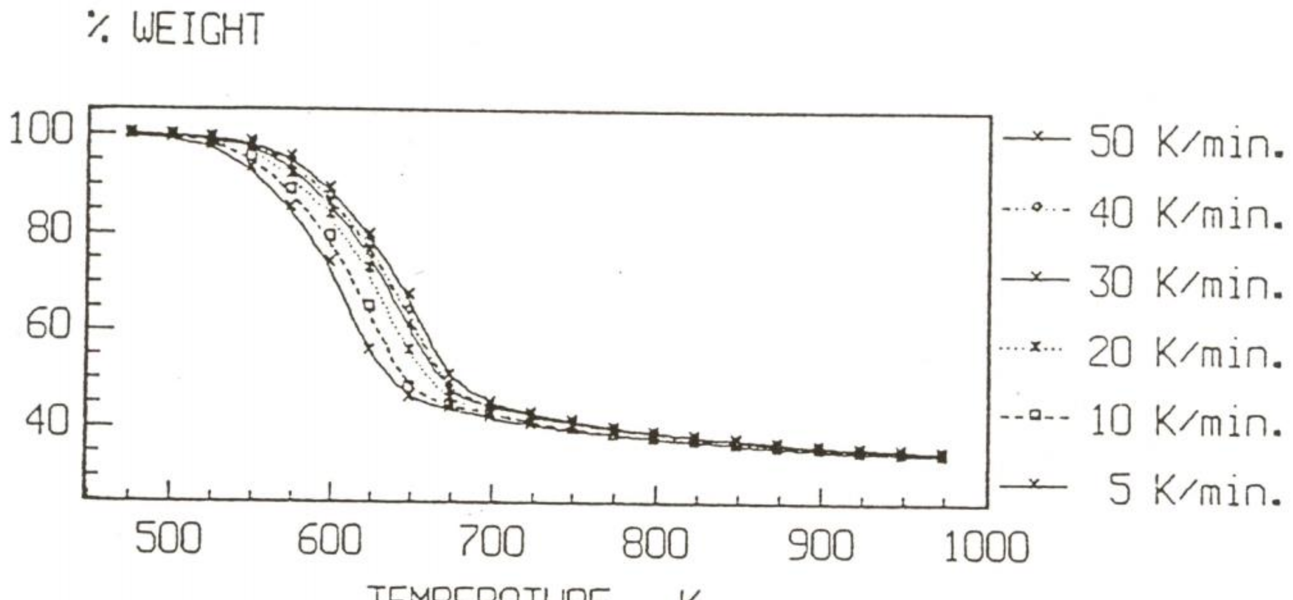


Figure 5 Effect of heating rate on rice husk pyrolysis.
Condition: sample mass = 2 mg. and particle size = 90-106 micron.,

characterize the pysolysis weight loss, as a result, can be reduced to two without greatly affecting the fit of the model[13]. The two parameters, E_o, the mean activation energy, and σ, the standard deviation, were estimated by using nonlinear Hooke and Jeeves optimizing method[14]. Details of the calculation were presented elsewhere[11]. The estimated values of E_o and σ for each heating rate are shown in Table 3 with the consistency of both parameters. The average values of E_o and σ are found to be 184.4 and 27.4 $kJ.gmol^{-1}$, respectively. Changing the value of k_o between 10^{11}-10^{15} s^{-1} showed insignificant effect on the values of both E_o and σ.

b.) Model based on main biomass components

The kinetic parameters and percentage of each main components used in the calculation were taken from Tables 1 and 2, respectively. The component defined as "others" (2.06%) was added to cellulose. The fourth order Runge Kutta algorithm was used to solve the set of differential equation as presented in Eqs. (9)-(13)[11].

Comparison between Experiment and Models

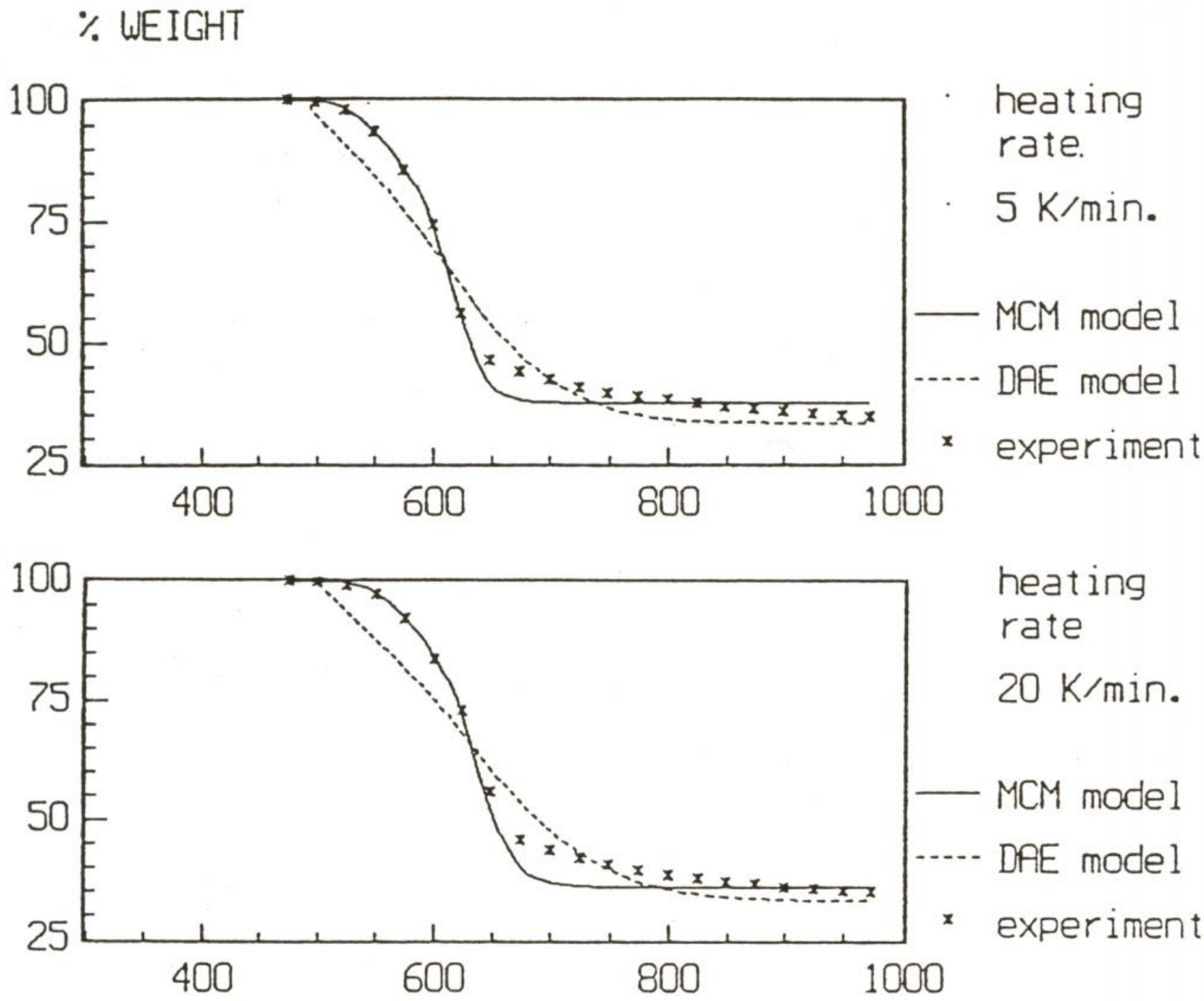
The predicted weight of rice husk sample from DAE and main biomass components models were compared with the non-isothermal TGA data at 5, 20, and 50 $K.min^{-1}$ heating rates as shown in Fig. 6. The DAE model showed fairly good agreement when compared to experimental data. This may be due to the assumption of pyrolysis reaction, which in fact consists of both parallel and series reactions, occurred only in the form of infinite parallel reaction. The main biomass components model, in contrast, showed good prediction without any adjustment of kinetic parameters. Testing this model with the other types of biomass undergoing pyrolysis is recommended in order to confirm its generalization. However, both models cannot predict the composition of evolved volatile, and thus are only suitable for combustion and char production processes.

Table 3 Kinetic parameters of rice husk pyrolysis obtained from the distributed activation energy model.

Heating rate ($K.min^{-1}$)	Eo $kJ.gmol^{-1}$	σ $kJ.gmol^{-1}$
5	185.3	25.62
10	182.7	25.62
20	184.5	27.30
30	185.1	28.56
40	184.2	28.56
50	184.5	28.77
Average	184.4	27.41

Isothermal Pyrolysis in Tube Furnace

The main biomass components model was used to predict the transient weight of whole rice husk pyrolyzed in an isothermal furnace, and the results obtained were compared with experimental data as depicted in Figure 7. Significant over prediction was observed when neglecting particle heat-up time was assumed in the model. To account for the heat transfer effect, both convection and radiative heat transfer rates from hot ambient gas to the rice husk particle were estimated using the overall heat transfer coefficient of 226-266 $W/m^2.K$. Details of calculation were presented elsewhere[11]. From heat balance, the particle temperature, neglecting its internal temperature gradient, at any given time can be calculated and was subsequently used to estimate the pyrolysis rate and particle weight. The results obtained, which are in good agreement with experimental data, are also shown in Fig. 7. The discrepancy may come from the inaccurate estimation of heat transfer coefficient and heat transfer surface area of the rice husk.



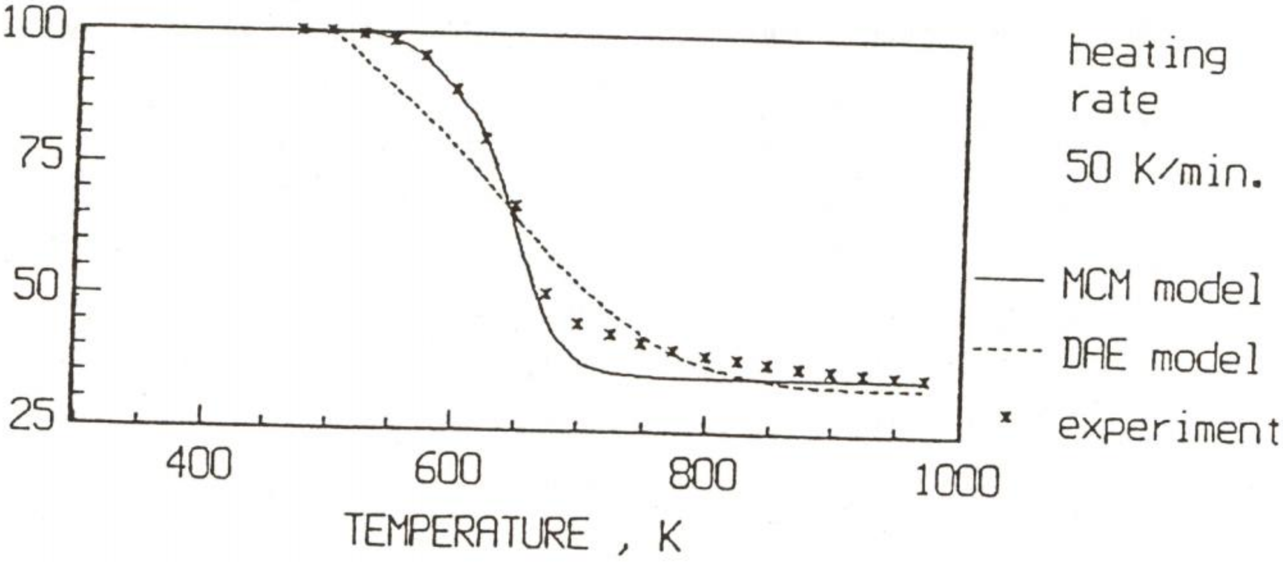


Figure 6 Comparison of rice husk weight during pyrolysis between experiment and models at different heating rates. Condition: sample mass = 2 mg and particle size = 90-106 micron. (MCM = main biomass components model and DAE = distributed activation energy model).

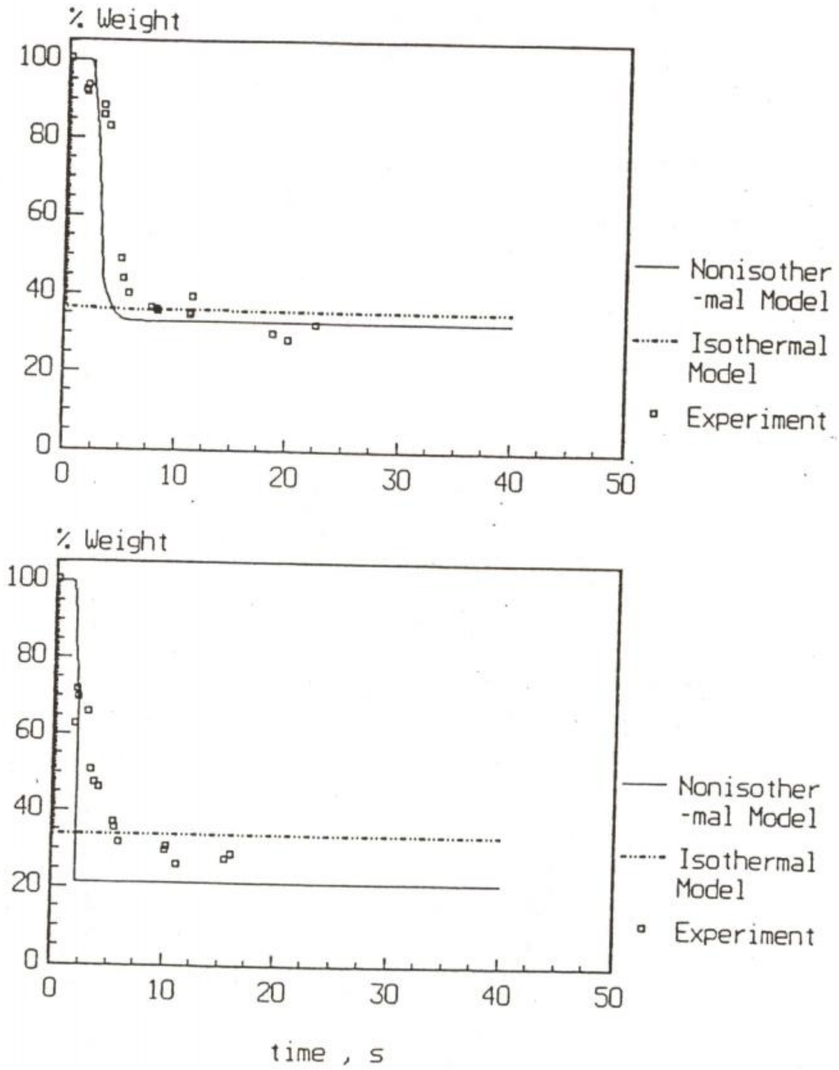


Figure 7 Comparison of rice husk weight between experiment and models during pyrolysis in a tube furnace at 1023 K (a) and 1123 K (b).

CONCLUSION

Using non-isothermal TGA technique with the sample mass below 7 mg, particle size below 150 micron, and heating rate below 50 K/min, the pyrolysis of rice husk is controlled by chemical decomposition reaction. The model based on main biomass components (cellulose, hemicellulose, and lignin) with a set of kinetic parameters proposed in the literature showed better prediction of sample weight history than the DAE model. When incorporated with the heat balance equation in which the intraparticle temperature gradient was neglected, this model enables fairly accurate prediction of single, whole rice husk pyrolysis.

NOMENCLATURE

B	weight fraction of virgin biomass
BI	weight fraction of intermediate
C	weight fraction of char
E	activation energy
k_o	frequency factor
n	reaction orde
q	heating rate
R	universal gas constant
T	absolute temperature
t	time
w	weight
x	fraction of weight loss
σ	standard deviation

Subscripts

f	final
i	volatile product or reaction order i
j	main biomass components: cellulose, hemicellulose, and lignin
o	initial

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ขอเชิญส่งบทความ

วิศวกรรมสาร ฉบับวิจัยและพัฒนา
(Research & Development Journal of the Engineering Institute of Thailand)

ในปัจจุบันนี้ประเทศไทยกำลังเร่งพัฒนาตัวเอง ทางด้านวิทยาศาสตร์และเทคโนโลยีสมัยใหม่ เพื่อรองรับการเจริญเติบโตของภาคอุตสาหกรรมในด้านต่าง ๆ ให้สามารถก้าวไปสู่ความเป็นประเทศพัฒนาทางอุตสาหกรรมสมัยใหม่ (Newly Industrialized Country) ได้ การพัฒนาวิทยาศาสตร์และเทคโนโลยีวิธีทำได้เร็วที่สุด คือ การซื้อเทคโนโลยีจากต่างประเทศ ดังเช่น ประเทศญี่ปุ่นในสมัยหลังสงครามโลก วิธีการนี้จะได้ผลเร็วเพราะไม่ต้องเสียเวลารอการวิจัย แต่ในขณะเดียวกัน นอกจากจะเป็นการสิ้นเปลืองเงินตราแล้ว ยังทำให้ประเทศนั้นต้องพึ่งพาประเทศอื่น ๆ อยู่ตลอดไป ประเทศที่ยอมซื้อไม่ว่าจะเป็นประเทศญี่ปุ่นจนมาถึงไต้หวัน เกาหลี และประเทศเพื่อนบ้านเรา คือ สิงคโปร์ในขณะนี้ ประเทศเหล่านี้ต่างมองการณ์ไกลเล็งเห็นถึงความสำคัญของการทำการวิจัยเพื่อพัฒนาวิทยาการต่าง ๆ ให้สามารถรองรับการขยายตัวของอุตสาหกรรมต่าง ๆ ได้ เพื่อจะได้ค่อย ๆ ปลดตัวเองจากภาระที่เกิดจากการเอารัดเอาเปรียบของประเทศที่เป็นเจ้าของเทคโนโลยีได้

ปัจจัยหนึ่งของการส่งเสริมการทำวิจัย คือ การมีวารสารวิจัยและพัฒนา มารองรับเพื่อเป็นการเผยแพร่ผลงานของนักวิชาการและนักวิจัยเอาไว้ ประโยชน์ที่มีนอกจากช่วยไม่ให้มีการทำวิจัยซับซ้อนกันแล้ว ยังช่วยให้เกิดความร่วมมือระหว่างนักวิชาการและนักวิจัยที่สนใจในปัญหาเดียวกัน เกิดมีการวิพากษ์วิจารณ์เพื่อให้ผลงานที่ติดตามมามีคุณภาพดียิ่งขึ้น วารสารทางวิชาการนี้จึงเป็นทรัพยากรทางความรู้ที่สามารถใช้เป็นเอกสารอ้างอิงในอนาคตสำหรับวิศวกร นักวิชาการ และนักวิจัยรุ่นหลังได้

วิศวกรรมสถานแห่งประเทศไทยฯ ได้เล็งเห็นความสำคัญของการมี "วารสารฉบับวิจัยและพัฒนา" จึงเห็นสมควรแยกเล่มออกมาจาก "วิศวกรรมสาร" (ซึ่งต่อไปจะเน้นไปทางสารคดีกึ่งวิชาการและข่าวสารปริทรรศน์มากกว่า) ต่อไปนี้วารสารวิชาการของ ว.ส.ท. จะออกเป็นราย 6 เดือน โดยจะรวมทุกสาขาวิชาทางวิศวกรรมศาสตร์เข้าด้วยกัน บทความทุกบทความที่ลงพิมพ์ในวารสารจะได้รับการประเมิน (Review) โดยกรรมการสาราณียกรและผู้ทรงคุณวุฒิในสาขาที่เกี่ยวข้อง เพื่อให้มั่นใจว่าทุกบทความที่ได้รับการลงพิมพ์ใน "วารสารฉบับวิจัยและพัฒนา" เป็นบทความที่มีคุณภาพและมีคุณค่ายิ่ง

การส่งบทความ เพื่อให้วารสารทางวิชาการมีมาตรฐานไม่ด้อยกว่าวารสารของสมาคมวิชาชีพทางวิทยาศาสตร์และเทคโนโลยีอื่น ๆ ทั้งในประเทศและต่างประเทศ คณะกรรมการสาราณียกรได้ให้ข้อแนะนำเพื่อผู้เขียนจะได้ใช้ในการพิจารณาส่งบทความมาลงตีพิมพ์ "วารสารฉบับวิจัยและพัฒนา" ดังนี้

1. บทความที่จะส่งควรเป็นผลงานการค้นคว้าวิจัยของผู้เขียนเอง และมีคุณค่าทางวิชาการพอสมควร
2. ภาษาที่ใช้อาจเป็นภาษาไทยหรือภาษาอังกฤษก็ได้ แต่ชื่อบทความ (title) ต้องมีทั้งภาษาไทยและภาษาอังกฤษ
3. เพื่อให้บทความมีความกะทัดรัดไม่เยิ่นย่อ และบทความควรมีความยาวไม่เกิน 10 หน้า กระดาษพิมพ์ดีด รวมทั้งรูปและตาราง
4. บทความต้องมีบทคัดย่อยาวไม่เกิน 15 บรรทัดพิมพ์ โดยมีทั้งภาษาไทยและภาษาอังกฤษ
5. ทุกบทความควรมีบทนำ (Introduction) บทสรุป (Conclusion) และเอกสารอ้างอิง (Reference)
6. เอกสารอ้างอิงถ้ามาจากเอกสารภาษาต่างประเทศ ให้เขียนเอกสารอ้างอิงนั้นด้วยภาษาอังกฤษ เอกสารอ้างอิงจะต้องประกอบด้วยชื่อผู้เขียน ชื่อบทความ ชื่อเอกสารที่ตีพิมพ์ สถานที่ตีพิมพ์ (ถ้าเป็นหนังสือ) ปีที่พิมพ์ และหน้าของบทความดังกล่าวในเอกสารนั้น)
7. ตารางและกราฟ ควรเป็นต้นฉบับที่ชัดเจน รูปถ่ายควรแนบฟิล์มมาด้วย คำบรรยายภาพควรเป็นภาษาอังกฤษ
8. โปรดส่งบทความมายังคณะกรรมการสาราณียกรฉบับวิจัยและพัฒนา ณ สำนักงานวิศวกรรมสถาน แห่งประเทศไทยฯ คณะวิศวกรรมศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย เขตปทุมวัน กทม. 10330 โทร. 218-6794-9, 251-2504 และ 250-1900
9. บทความทุกบทความจะได้รับการประเมินเพื่อตีพิมพ์โดยผู้ทรงคุณวุฒิในสาขาวิชาเดียวกัน
10. คณะกรรมการสาราณียกร อาจให้ผู้เขียนปรับปรุงบทความให้มีเหมาะสมยิ่งขึ้น และทรงไว้ซึ่งสิทธิในการตัดสินลงพิมพ์บทความหรือไม่ก็ได้
11. ผู้เขียนบทความเป็นผู้รับผิดชอบต่อผลทางกฎหมายใด ๆ ที่อาจเกิดขึ้นเนื่องมาจากบทความนั้น ๆ

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