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INVESTIGATION OF THERMAL DECOMPOSITION OF RICE HUSK

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ABSTRACT

Studies on the thermal decomposition of untreated rice husk and that treated with HCl and H_2SO_4 of various concentrations were carried out by TG, DTG and DTA. The mass loss occurred in three distinct stages, namely, removal of moisture, release of volatile matter and burning of combustible material. The corresponding temperature ranges for untreated husk were 40–150, 215–350 and 350–690°C. The final temperature of combustion decreased with acid-treatment of the husk. The thermal decomposition of the husk was found to be an exothermic process.

INTRODUCTION

Husk is a major by-product of the rice milling industry. Its utility as a valuable product has always remained a problem. The carbon-free ash (white ash) obtained from rice husk has opened a new dimension in its utilisation concept during recent years [1–3]. White ash, as a source of high-grade amorphous silica, can be utilised for the production of silicon [4–7]. The production of amorphous white ash involves controlled combustion of the husk at low temperatures [8,9]. It has been reported that acid leaching of the husk helps in the removal of most of the metallic impurities [10] for the production of white ash. Earlier work [11] also indicates the nature of the thermal decomposition of acid-treated ground husk by using thermogravimetry (TG). The thermal decomposition occurred in three main stages, namely, drying, removal of organic volatile matters and fixed carbon. Some intermediate stages of inappreciable mass loss were also observed. However, the above study did not include any information on other thermal analyses, like derivative thermogravimetry (DTG) or differential thermal analysis (DTA).

In view of the above, the present study was undertaken with the following objectives:

(1) to study the thermal decomposition of rice husk by TG, DTG and DTA;

(2) to study the effect of leaching rice husk in HCl and H_2SO_4 of different concentrations on its thermal decomposition.

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EXPERIMENTAL DETAILS

Materials preparation

Rice husk, obtained from a high-yielding variety of paddy, namely, Mashuri was thoroughly wet-cleaned, dried and then ground to 40 mesh.

The ground samples were leached in different concentrations of HCl (1, 3 and 5 N) and H_2SO_4 (4.5 and 9 N) at $50 \pm 2°C$ for 2 h. After leaching, the husk was thoroughly washed with distilled water and then dried.

Thermal analyses

The TG, DTG and DTA of the untreated and acid-treated husks, as mentioned above, were performed using thermal analyser (Mettler TA 2000 system) under static ambient conditions. Platinum crucibles and heat-treated α-Al_2O_3 were used as sample holders and reference sample, respectively. The

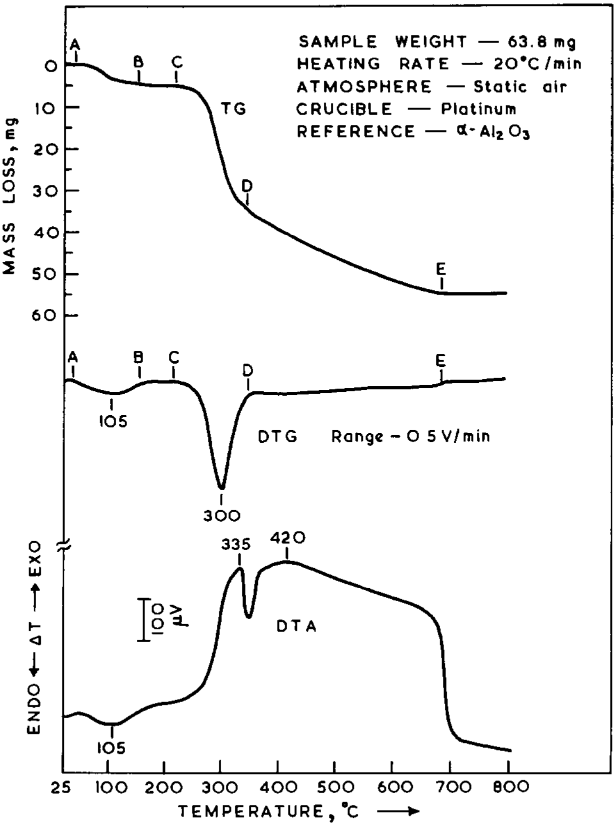

Fig. 1. TG, DTG and DTA curves for rice husk (40 mesh).

furnace temperature programme was 20°C min^{-1} from an ambient temperature of 25°C until a constant weight was attained. The TG, DTG and DTA curves were recorded simultaneously along with temperature rise.

RESULTS AND DISCUSSION

The simultaneous TG, DTG and DTA curves for untreated and various acid leached husk samples are shown in Figs. 1–6. The nature of the TG curves indicated that the loss of mass occurred in three major stages. One intermediate stage of inappreciable mass loss was also observed after the first stage. This result is in good agreement with previous work [11]. The various stages of decomposition and the temperature range for each stage were identified from the trend of the TG and DTG curves. The four different stages are marked in the figures as A–B, B–C, C–D and D–E. The

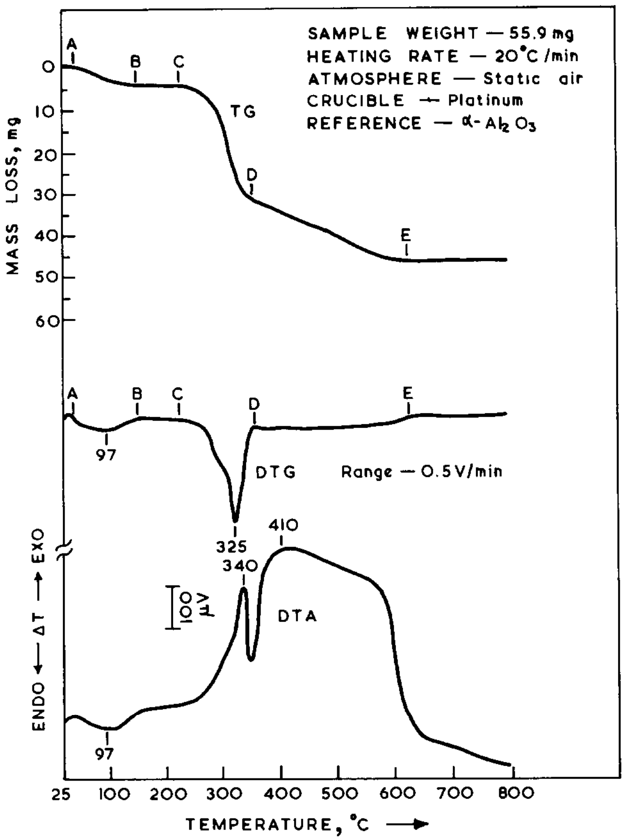

Fig. 2. TG, DTG and DTA curves for rice husk (40 mesh) treated with 1 N HCl at 50°C for 2 h.

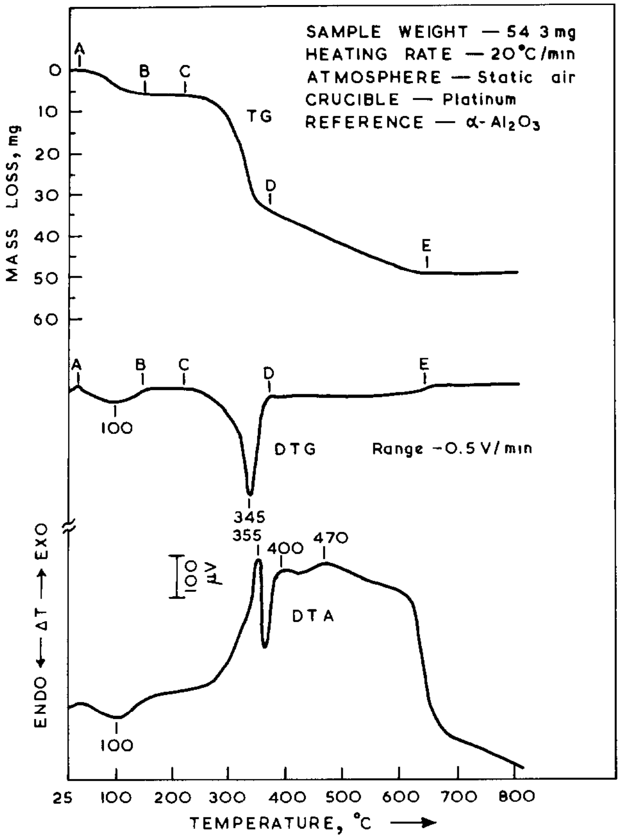

Fig. 3. TG, DTG and DTA curves for rice husk (40 mesh) treated with 3 N HCl at 50°C for 2 h.

temperature ranges are given in Table 1. The percentage and rate of mass loss in each stage as calculated from the TG curves are indicated in Table 2. Table 3 shows various peak temperatures in the DTG and DTA curves.

TABLE 1

Temperature range for different stages of mass loss in the TG curves

Treatment	Temperature range (°C)			
	Stage I	Stage II	Stage III	Stage IV
Untreated	40–150	150–215	215–350	350–690
1 N HCl treated	40–150	150–225	225–355	355–630
3 N HCl treated	40–150	150–225	225–375	375–650
5 N HCl treated	40–150	150–250	250–375	375–650
4.5 N H_2SO_4 treated	40–150	150–225	225–350	350–645
9 N H_2SO_4 treated	40–150	150–230	230–375	375–675

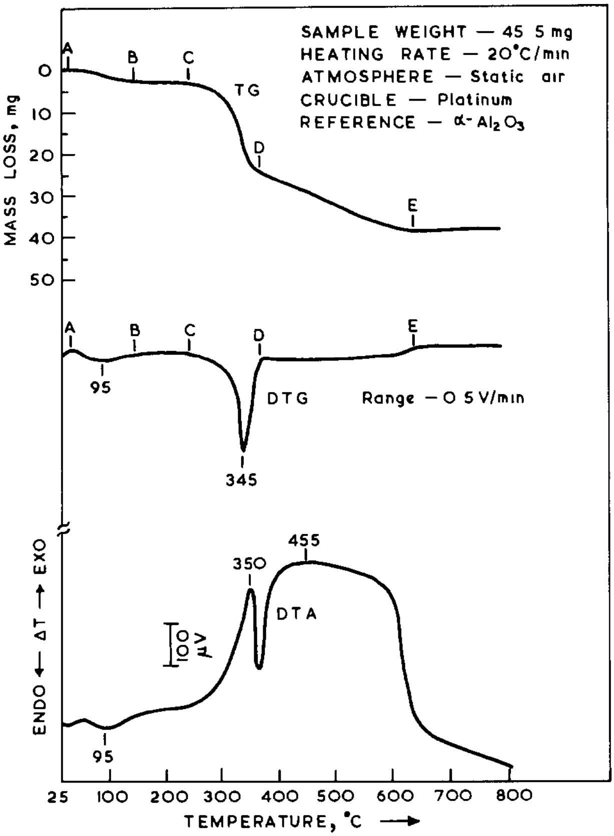

Fig. 4. TG, DTG and DTA curves for rice husk (40 mesh) treated with 5 N HCl at 50°C for 2 h.

The TG curves show that the mass loss in the first stage took place in the range 40–150°C. The DTG curves exhibit a peak in the range 75–105°C which corresponds to the maximum rate of mass loss. The DTA curves also

TABLE 2

Percentage (%) and rate (mg min^{-1}) of mass loss in different stages

Treatments	Stage I		Stage II		Stage III		Stage IV	
	Percent	Rate	Percent	Rate	Percent	Rate	Percent	Rate
Untreated	8.15	0.9	0.95	0.19	48.0	4.56	30.5	1.14
1 N HCl treated	8.94	0.9	0.35	0.05	48.0	4.15	28.0	1.14
3 N HCl treated	11.40	1.0	1.10	0.17	51.5	3.90	29.0	1.14
5 N HCl treated	7.05	0.8	0.90	0.07	48.5	3.52	32.0	1.05
4.5 N H_2SO_4 treated	4.70	0.5	0.40	0.06	51.0	3.66	28.5	0.95
9 N H_2SO_4 treated	6.60	0.9	0.75	0.09	50.6	3.70	31.0	1.05

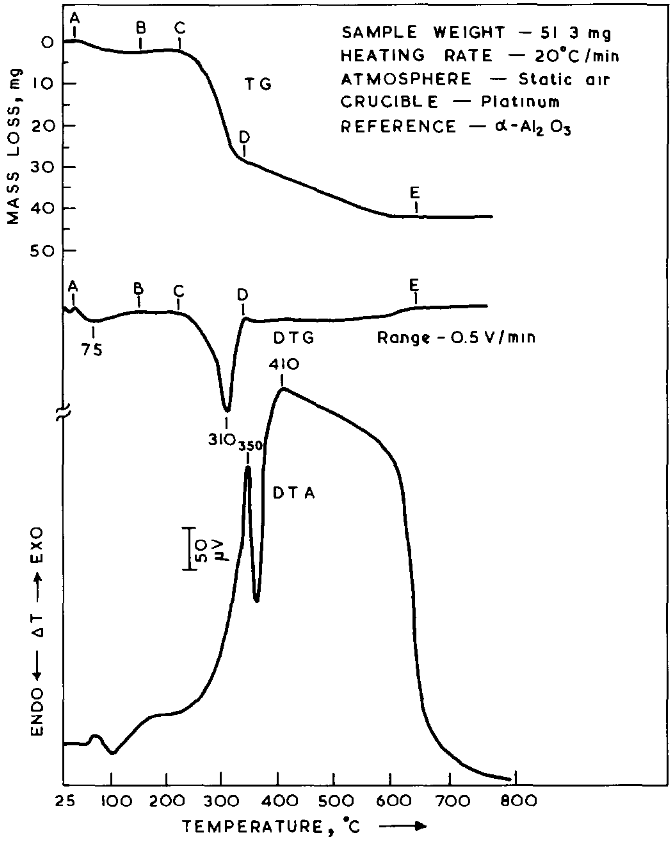

Fig. 5. TG, DTG and DTA curves for rice husk (40 mesh) treated with 4.5 N H_2SO_4 at 50°C for 2 h.

indicate an endothermic peak around 100°C. The mass loss ranging from 4.7 to 11.4% associated in this stage may be attributed to the removal of moisture from the material. It was also reported earlier [12] that the thermal

TABLE 3

Peak temperatures (°C) in DTG and DTA curves corresponding to different stages of mass loss

Treatments	Stage I		Stage II		Stage III		Stage IV	
	DTG	DTA	DTG	DTA	DTG	DTA	DTG	DTA
Untreated	105	105 endo	—	—	300	335 exo	—	420 exo
1 N HCl treated	97	97 endo	—	—	325	340 exo	—	410 exo
3 N HCl treated	100	100 endo	—	—	345	355 exo	—	400 exo 470 exo
5 N HCl treated	95	95 endo	—	—	345	350 exo	—	455 exo
4.5 N H_2SO_4 treated	75	100 endo	—	—	310	350 exo	—	410 exo
9 N H_2SO_4 treated	100	100 endo	—	—	340	350 exo	—	435 exo

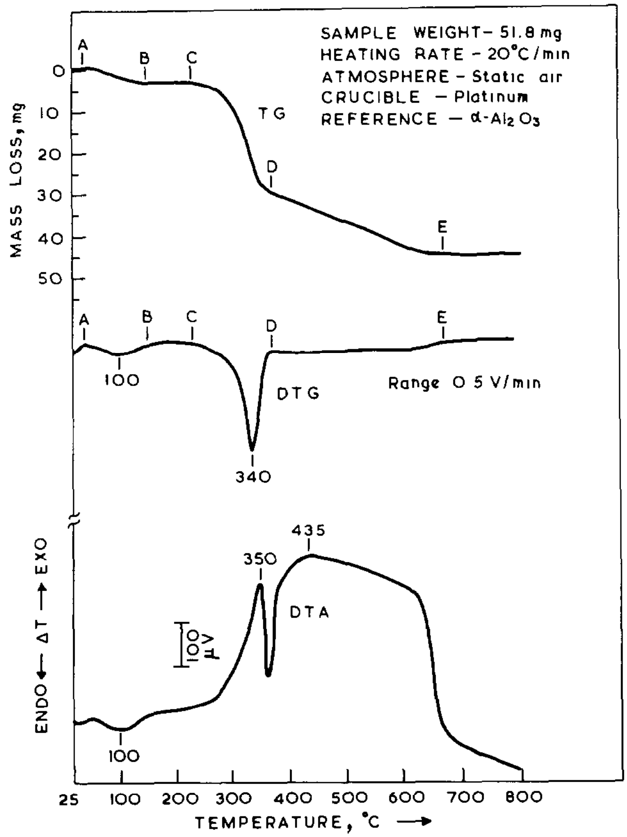

Fig. 6. TG, DTG and DTA curves for rice husk (40 mesh) treated with 9 N H_2SO_4 at 50°C for 2 h.

decomposition of oven-dried husk was negligible below 200°C.

The second stage was identified as a plateau in the TG curves in the range 150–250°C with an inappreciable mass loss of 0.35–1.1%, while the DTG and DTA curves do not show any peak in this stage. This may be considered as a transition stage.

The third stage of decomposition took place in the range 215–375°C indicating a steep fall and a sharp peak in the TG and DTG curves, respectively. The mass loss associated with this stage (48–51.5%) may be due to the removal of volatile matter [12]. The rate of mass loss for untreated husk was higher than that for the treated husk. The maximum rate of mass loss as indicated by the DTG curves was found to be dependent on the concentration of the acid-treatment of the husk. For untreated husk it was around 300°C and for HCl and H_2SO_4 treatment it was within the ranges 325–345 and 310–345°C, respectively. The DTA curves exhibit exothermic peaks in the range 335–355°C.

The fourth stage of mass loss (28–32%) in the range 350–690°C corresponds to the combustion process. It is interesting to note that the final temperature in this stage is higher for untreated husk than that for the treated husk. For untreated it was 690°C and for treated husk it varied from 630°C for 1 N HCl treatment to 650°C for 5 N HCl treatment, and 645°C for 4.5 N H_2SO_4 treatment to 675°C for 9 N H_2SO_4 treatment. This shows that treatment with acids of lower concentration is desirable. The DTG curves show a gradual decrease in the rate of mass loss and the DTA curves indicate an exothermic reaction.

Among the four stages, the major decomposition occurred in the third and fourth stages. The temperature range of the fourth stage was higher and the rate of mass loss was slower as compared to the third stage.

CONCLUSIONS

(1) The TG analysis of untreated and acid-treated rice husk revealed three distinct stages of mass loss, namely, removal of moisture, release of volatile matter and burning of combustible material.

(2) The removal of moisture from untreated and acid-treated husk took place at temperatures ranging from 40 to 150°C.

(3) The volatile matter was released from untreated and acid-treated husk in the ranges 215–350°C and 225–375°C, respectively.

(4) The combustion of untreated husk took place at temperatures ranging from 350 to 690°C. The final temperature of combustion in this stage decreased with acid-treatment of the husk.

(5) The DTA records exhibit an exothermic reaction during the course of thermal decomposition and an endothermic peak during the removal of moisture.

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