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Thermogravimetric analysis of paddy straw

S.KALEEMULLAH
Department of Agricultural Processing, Tamil Nadu Agricultural University, Coimbatore - 641 003

Abstract : Results of the studies on thermogravimetric analysis (TGA) of raw and IN HCl acid treated ground paddy straw revealed that thermal degradation takes place in three main stages of mass loss, namely (1) removal of moisture (2) removal of volatile matter and (3) oxidation of fixed carbon. The rate of removal of volatile matter was faster in the untreated sample than that in the acid treated sample. But the rate of removal of fixed carbon was faster in acid treated paddy straw than that untreated paddy straw. Temperatures of above 340 and 375oC are required to remove fixed carbon from raw and IN HCl treated paddy straw to get amorphous silica. Hydrochloric acid leaching of paddy straw at 75oC for one hour prior to combustion is necessary for production of ash of milky white colour. *(Key Words:* Paddy Straw, Thermogravimetric Analysis, Ash, Silica)

Paddy crop being one of the major cereal crops of the world produces major byproduct, namely, paddy straw. It is estimated that India produces about 120 million tons of paddy straw every year (Sarnpathrajan et al., 1999). Paddy straw with a low bulk density (25.42 kg/m^3) (Kaleemullah, 1988) and high silica content has some traditional use as cattle fodder. Paddy straw, a fibrous material, therefore, poses disposal problems wherever it is produced in excess. However, in recent years, as the concept of 'converting waste into wealth' became more popular, serious research work is in progress to use the paddy straw in a meaningful way. The suggested uses are based either on its organic constituents or on the silica content. Recently, paddy straw silica has been identified as a unique source of high-grade amorphous silica. The paddy straw contains 11-15% silica and that of paddy straw ash contains 69.1-76.8% silica (Juliano, 1985).

The silica present in paddy straw being of biogenic origin is inherently amorphous. It was reported that the rice husk silica would be transformed into a more stable crystalline form when it is subjected to a temperature above 720°C (Singh et al., 1981; Hamad and Khattab, 1981). The amorphous silica obtained from controlled combustion of paddy straw at lower temperature is chemically more reactive and useful than crystalline silica. Amorphous silica can be used in various industries like glass, rubber and ceramics. The paddy straw silica could be an attractive raw material for the production of pure silicon, which is essentially used in photovoltaic and semiconductor industries for manufacturing solar cells, transistors, integrated circuits and microchips. It was reported that the acid leaching of rice husk helps in the removal of most of the metallic impurities for the production of amorphous silica (Mishra *et al.* 1985).

Keeping the above points in view, a study was undertaken to understand the thermal degradation of untreated and 1N HC1 acid treated paddy straw by thermogavimetric analysis. By conducting thermogravimetric analysis, the minimum temperature required to remove moisture content, volatile matter and fixed carbon can be determined.

Materials and Methods

The paddy straw was chopped to one centimeter and was thoroughly wet cleaned and dried. The dried material was leached with 1N HC1 at 75°C for one hour to remove metallic impurities. The leached material was washed with tap water five times to remove the HCI. Finally, it was thoroughly washed with distilled water to remove all the impurities and the last traces of HCI. The wet material was dried in sunlight so that its moisture content reduced to 7.5%(wb). The raw and acid treated ground samples (40 mesh) were used for themogravimetric analysis (TGA).

The TGA of paddy straw was carried out using a thermogravimetric analyzer (Fertilizer Corporation of India Ltd., Ranchi). The sample was taken in a small platinum crucible and its initial weight was recorded. The sample inside the furnace was heated at a constant rate of heating (1°C/min) by controlling the input power with the help of an autotransformer. During the entire heating operation, the mass loss of the material under investigation was recorded at an interval of 2.5 minutes. The sample was heated from ambient to 750°C of the furnace temperature.

Results and Discussion

The TGA curves of paddy straw representing the mass loss as a function of temperature are shown in Fig. 1 When paddy straw was heated at 10°C/min heating rate, the mass loss was in three major stages excluding the transition stage after the drying stage. The three major stages during decomposition were: 1) remval of moisture

Table 1. Details of thennogravimetric analysis of ground rice straw at 10°C/min heating rate

Condition of straw	Decay stage	Temperature range (°C)	Mass loss (%wb)	Time (min)	Rate of mass loss (%/min)	Colour of ash
Untreated	1	25-150	11.00	12.5	0.88	
	11	150-200	1.25	5.0	0.25	
	III	200-340	67.75	14.0	4.84	
	IV-a	340-500	10.50	16.0	0.66	Blackish
	IV-b	500-750	2.50	25.0	0.10	white
1N HCl treated	I	25-125	7.00	10.0	0.70	
	II	125-175	1.00	5.0	0.20	
	III	175-375	64.00	20.0	3.20	
	IV-a	375-500	15.00	12.5	1.20	Milky
	IV-b	500-750	3.25	25.0	0.13	white

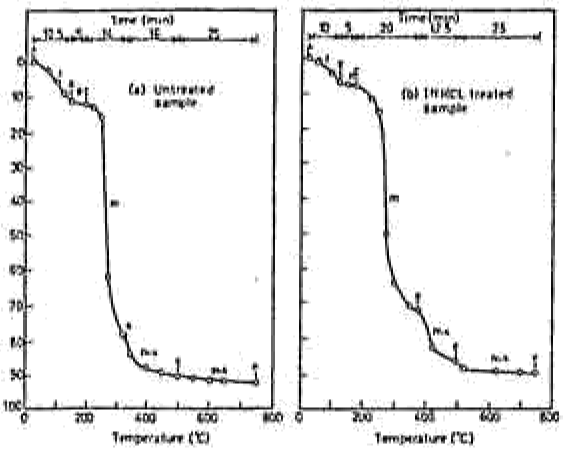

Fig. TGA Curves of ground paddy straw at 10°C/min heating rate

(drying) 2) removal of volatile matter and 3) oxidation of fixed carbon, The last stage had two substages. All these stages were marked in the Fig.1 as A-B, B-C, C-D, D-E and E-F, which were identified from the trend of the TGA curves. The lad stage had two sub stages indicated as IV-a and IV-b. The temperature ranges for the various stages of mass loss and the rates of mass loss for 10°C/min heating rate are given in Table 1.

The TGA curves of untreated and acid treated paddy straw at 10°C/min heating rate show that the temperature ranges of the same stages were different for untreated and acid treated straw. In the first stage, the temperature range was 25-150°C for untreated and 25-125°C for acid treated sample. The rates of moisture loss were 0.88 and 0.7%/min in untreated and acid treated samples respectively. A mass loss of 11 and 7% respectively occurred in this stage corresponding to the removal of moisture from the material.

In the second stage, the temperature range was 150-200°C in untreated sample and 125-175°C in acid treated one. The mass loss in this stage was 1.0-1.25% which was very less compared to other stages. This stage was considered as a transition stage as it might be due to the loss of the last trace of moisture, CO_2 gas etc. The rate of mass loss in this stage was 0.2-0.25%/min. The major part of the mass loss took place in the third stage. The sharp fall of the TGA curves as shown in Fig. 1, in the temperature ranges of 200-340°C in untreated sample and 175-375°C in acid treated sample was due to the removal of volatile matter.

The mass loss of volatile matter was about 67.75% in the untreated sample and 64% in treated one. It appears that some percentage of volatile matter had been leached away when the straw was leached with 1N HC1 at 75°C and washed away with distilled water. The rates of mass loss of volatile matter were 4.84 and 3.2%/min in the untreated straw and treated straw respectively. It implies that the rate of removal of volatile matter was faster in the untreated sample than that in the acid treated sample due to the presence of more volatile matter in the untreated one.

The fourth stage of the TGA curves, which showed a gradual decrease in the mass was due to a slow combustion of fixed carbon. Initially, the combustion rate was high but after some time, the rate drastically fell down. The temperature ranges of the substage IV-a were 340-500° and 375-500°C and rates of mass loss of fixed carbon were 0.66 and 1.2%/ min for the untreated and add treated straw respectively. It reveals that the rate of removal of fixed carbon was faster in the case of treated straw than that in the untreated straw. The rate of removal of fixed carbon in substage IV-b was only 0.1-0.13% / min. It clearly indicates that the removal of fixed carbon was faster in the first substage and very slow in the second substage. The amount of ash obtained was 7 and 9.75% (wb) from untreated and 1N HC1 treated straw. The reason for obtaining more quantity of ash in the case of treated straw may be due to the leaching of volatile matter when the straw was treated with 1N HC1.

From Table 1, it is clear that temperatures of above 340 and 375°C are required to remove fixed carbon from raw and 1N HC1 treated paddy straw to get amorphous silica. The colour of the ash obtained after thermogravimetric analysis was blackish white in the case of untreated straw and milky white in the case of 1N HC1 treated straw. It may be due to the removal of the most of the metallic impurities in acid treated paddy straw.

Conclusions

* Bose, T.K., Som,M.G and Kabir,J. (1993). *Vegetable crops*. Naya Prakash Publications, Calcutta, p.20.The thermal decomposition of untreated and acid treated paddy straw takes place in three major stage of mass loss namely (a) removal of moisture (drying) (b) release of volatile matter (devolatilization) and (e) oxidation of fixed carbon.
* The TGA curves show that the temperature ranges of the same stages were different for untreated and add treated paddy straw.

* The rate of removal of volatile matter was faster in the untreated paddy straw than that in the acid treated sample due to the presence of more volatile matter in sample the untreated one.
* The rate of removal of fixed carbon was faster in the acid treated paddy straw than that in untreated one.
* The rate of removal of fixed carbon was faster in the first stage and very slow in the second substage.
* The colour of the ash obtained after thermogravimetric analysis was blackish white in the case of untreated straw and milky white in the case of acid treated straw. This may be due to the removal of the most of the metallic impurities in acid treated straw.
* The temperatures of above 340 and 375oC are required to remove fixed carbon from raw and IN HCI treated paddy straw to got amorphous silica.

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Combining ability analysis for biological nitrogen fixation traits under controlled conditions in greengram *Vigna radiata* (l.) Wilczek.

K. BHADRA AND D. WILSON

Dept. of Plant Breeding and Genetics, College of Agriculture, Vellayani, Thiruvananthapuram-695 522.

Abstract : Combining ability analysis for different biological nitrogen fixation traits in greengram (*Vigna radiata* (L.) Wilczek) was carried out under controlled conditions. Seven parental varieties and their F_1 hybrids were analysed for the estimation of combining ability and gene action under rhizobium inoculated, non-inoculated and absolute control situations. Analysis of variance revealed significant mean square due to general combining ability (gca) and specific combining ability (sca). The parents showed significant variation with respect to gca effect. The parent NDM-88-14 is identified as the best parent having significant gca effect for all the biological nitrogen fixation traits. The hybrids showed sca effects at varying levels. ***(Key words:** Greengram, Biological nitrogen fixation, Combining ability).*

Pulses are the major source of dietary protein. Legume - Rhizobium symbiosis play a predominant role in agriculture because they can fix atmospheric nitrogen. Sarawgi *et al.* (1999) reported enhanced grain yield in chickpea following rhizobial inoculation. Chaudhary *et al.* (2000) observed enhanced dry matter production in greengram at different growth stages as a result of rhizobial inoculation. Xu Qiaozhen *et al.* (2000) revealed that combatibility of soybean cultivars with rhizobial strain is controlled by a pair of dominant genes. Greengram (*Vigna radiata* (L.) Wilczek) is one of the important grain legume crops of India and is cultivated in upland and in summer rice fallows. There is possibility of breeding legumes for increased nitrogen fixation by improving the biological nitrogen fixation traits (Singh and Murthy, 1988). For developing varieties having good nitrogen fixing capacity and reasonable yield the information on combining ability of parents and the nature of gene action involved in the inheritance of biological nitrogen fixation traits is very essential.