COMPARATIVE ANALYSIS OF MORPHOLOGICAL AND STRUCTURAL CHANGES IN GAMMA AND ELECTRON BEAM IRRADIATED SUGARCANE BAGASSE

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Sugarcane bagasse is an abundant source of cellulose and hemicelluloses that could be hydrolyzed to yield fermentable sugars, which can be utilized for the production of biofuel and other high-value bio-chemicals. To do so, it has to be made accessible for hydrolyzing chemicals and enzymes, and radiation exposure is one of the most effective and green techniques, among other physico-chemical processes. The present study investigated the effects of gamma and electron beam (e-beam) irradiation on sugarcane bagasse, with respect to changes in its physical, chemical, thermal and morphological characteristics. Sugarcane bagasse was irradiated with gamma radiation, using Co^{60} at a dose rate of 2.5 kGy/h, and electron beam at a dose rate of 2.5 kGy/pass. The maximum dose was varied up to 1000 kGy and changes in the physico-chemical characteristics of bagasse were observed at 500 kGy dose exposure. The physical appearance of bagasse (after gamma and e-beam treatments) changed from off-white to yellow in colour, while beyond 500 kGy, the samples became fluffy. With an increase in the radiation dose, the cellulose content reduced from 48% to 36%, following Co^{60} gamma exposure, and to 16% after e-beam exposure at 1000 kGy. The hemicellulose content was found to reduce from 31% to 16% after 1000 kGy of Co^{60} gamma exposure, but after e-beam radiation, it increased to 39%. The lignin fraction did not change much after any of the treatments, and was found to be in the range of 19-20% and 17-23%, after gamma and e-beam radiation exposure, respectively. In most of the irradiated samples, X-ray diffraction (XRD) confirmed a significant increase in crystallinity index with the increase in the radiation dose up to 1000 kGy. However, a decrease in the crystallinity index of bagasse was observed after e-beam irradiation. Scanning electron microscopy (SEM) analysis showed remarkable disruption of the structure, caused by high energy irradiations (gamma and e-beam). The particle size analysis indicated fragmented particles on increasing irradiation doses, but the distribution is more prominent in the case of the e-beam treatment. A lowering of the derivative thermogravimetric (DTG) peak from 339 °C in raw bagasse to 295 °C and 303 °C, for the samples subjected to gamma and e-beam radiation, respectively, was observed, in the thermal study of the biomass. The physico-chemical changes observed during the study clearly indicated that ionizing radiation exposure of lignocellulosic biomass led to the disintegration of its matrix, which may give easy access to hydrolytic chemicals or enzymes. Thus, it can be concluded that, although both ionizing radiations investigated here can fulfill the objective of disintegrating the biomass structure, gamma is more effective than e-beam radiation.

Keywords: biomass, electron beam irradiation, gamma, sugarcane bagasse, ionizing radiation

INTRODUCTION

Lignocellulose is one of the most widespread materials known globally, but its valorization in various areas involves high pretreatment costs. It is a highly organized complex plant material, composed mostly of semi-crystalline cellulose, amorphous hemicelluloses and lignin. Physical and/or chemical pretreatments are necessary to reduce crystallinity, disrupt the hydrogen bonding of cellulose and make it accessible to hydrolytic chemical/enzymatic reactions.[1]

With recent advancements in radiation technology, both electron beam and gamma irradiation have become worthy tools, showing some advantages over other pretreatment

methods. Ionizing radiation is known to degrade materials and can be applied as a pretreatment method in the process of ethanol production from biomass.[2] The objectives of the pretreatment process is to break down the lignin structure and disrupt the crystalline structure of cellulose, so that the acids or enzymes can easily access and hydrolyze the cellulose. The pretreatment can be the most expensive process in the conversion of biomass to fuels however, but it has great potential for improvements in efficiency and lowering of costs.[3-8,36]

The use of high energy radiation methods, including γ-ray, ultrasound, e-beam, UV and microwave heating, can ease the digestibility of lignocellulosic biomass using acid and enzymatic hydrolysis of lignocelluloses.[9-14] Amongst the methods applied so far as pretreatment of lignocellulose, irradiation with gamma or electron beam are considered as dry and green processes that can cause the desired changes in its chemical and physical properties.[15-17] The high energy radiation can affect lignocellulosic biomass, causing an increase in specific surface area, a decrease in the degree of polymerization and crystallinity of cellulose, resulting in easy hydrolysis of hemicelluloses, and partial depolymerization of lignin.[18] Gamma and e-beam irradiation methods are considered as dry and green physical pretreatment processes, as they do not involve any use of chemicals.

Ionizing radiation can originate either from a radioactive source or from an electron accelerator. Both are capable of replacing chemical treatments for the modification of polymer ingredients present in the lignocellulosic matrix.[19] The effects of e-beam irradiation on cellulose have been evaluated earlier.[20-22] Chain scission and reduced crystallinity have been identified as the most prominent effects. It has been also revealed that using e-beam radiation of 1.5 MeV, with doses from 5 to 100 kGy, affected the sugarcane bagasse structure, causing some lignin and cellulose cleavage.[23-24] Other studies have also confirmed that ionizing irradiation is equivalent to other methods in terms of making the lignocellulosic matrix easily digestible.[25-29]

The present study aims to analyze the effects of gamma and electron beam irradiation on sugarcane bagasse, with respect to changes in its physical, chemical, thermal and morphological characteristics.

EXPERIMENTAL

Materials

Sugarcane bagasse was procured from M/s Simbhaoli Sugar Mill, Simbhaoli, UP, India. Sodium hydroxide, sulphuric acid, nitric acid, hydrochloric acid, acetic acid, ethanol *etc*. for characterization of bagasse were of LR grade and purchased from Finar and Fischer Scientific, India. The samples were packed in aluminium foil packets and exposed to gamma radiation at Shriram Applied Radiation Centre (SARC), Delhi, and to e-beam at the Board of Radiation and Isotope Technology (BRIT), Vaashi, Mumbai.

Characterization of bagasse

The moisture content of the bagasse was determined as per the method of IS 7874,[30] by measuring the loss in weight upon drying at 105 ± 2 °C for 4 h until constant weight was achieved. Bulk density was determined as per IS 33, by introducing the sample into a 500 mL measuring cylinder, while tapping upside down for 60 times.[31]

The cellulose content was measured as per the method described in an earlier study.[32] To 1 g of oven dried bagasse, an amount of 15 mL of 80% aq. acetic acid and 1.5 mL of concentrated nitric acid were added and refluxed for 20 min. The filtrate was washed with hot water, dried and weighed. The dried material was then incinerated in a muffle furnace at 550 °C for 5 h and then weighed to estimate residual ash, by subtracting the weight of ash with dried filtrate weight.[32]

The hemicellulose content was determined as per the method described by Youn *et al*.[26] A 10 g of bagasse sample was autoclaved with 100 mL of 1 N NaOH at 121 °C for 1 h. The insoluble fraction of bagasse was neutralized with hydrochloric acid. The hemicellulose was filtered, washed, dried and weighed to estimate the hemicellulose content. The dissolved fraction of hemicelluloses present in the filtrate was precipitated by adding 95% ethanol in the solution. The precipitate was filtered, washed, dried and weighed (hemicellulose content).[26]

The lignin content of sugarcane bagasse was determined as per ASTM D1106. Approximately 2 g of bagasse sample was taken and 30 mL of cold 72% H_2SO_4 was added. The mixture was diluted by adding 560 mL of distilled water, and refluxed for 4 h. The material was filtered using a G-4 sintered glass crucible. The residue was washed and dried to determine the lignin content. The dried lignin was weighed in a platinum crucible and ignited in a muffle furnace at 900 °C for 2 h to estimate the ash content.[33]

Gamma radiation and dosimetry studies

The gamma irradiation of the samples was performed at the Shriram Applied Radiation Centre (SARC), located at SRI, Delhi, India. The irradiation unit uses radioactive isotope Co^{60} as the irradiation

source. The energy source at the gamma irradiation facility is of 800 kCi, with a capacity of 25 kGy exposure rate in 7 h. The irradiator is a panoramic, pool type, in which the source is contained in a storage pool of water and is fully shielded when not in use.

The sugarcane bagasse was exposed to gamma irradiation at different dose levels, *i.e.* 50, 100, 500 and 1000 kGy. The quantity of dose absorbed by the material was measured using ceric-cerous dosimeter standards, in accordance with the guidelines of ISO/ASTM E 51205.[34] The dosimeter was placed along with the sugarcane bagasse samples exposed to radiation. After completion of the gamma radiation processing of bagasse samples, the absorbed dose was measured and recorded in each batch of the irradiated samples. The uncertainty of the radiation dose given by the system to the sample was measured as ± 10%. The effect of gamma radiation exposure was studied with respect to the changes occurring in the physical, chemical and surface properties of the sugarcane bagasse. The schematic diagram of the gamma irradiation unit using Co^{60} is depicted in Figure 1.

Electron beam irradiation

Electron beam (e-beam) radiation exposure was performed at the Board of Research in Isotope Technology (BRIT), Vaashi, Mumbai, at dose levels of 50, 100, 500 and 1000 kGy, with a dose rate of 2.5 kGy/second/pass. The e-beam accelerator is of high power, ILU-6-M3 type, produced by the Institute of Nuclear Physics, Novosibirsk, USSR. The source of electrons was Lanthanum Hexa Borate. The e-beam accelerator has beam energy of 2 MeV and power of 20 kW, penetration depth with 10 mm in unit density. The conveyer speed was 3 cm/second. The radiation/pass was of 2.5 kGy (time approx. 1 min). The irradiation window has 80 cm length and 8 cm width. The distance between the sample and the window is 25 cm. FTR-125, Radiochromic and CTA film dosimeters were used in the study. A schematic diagram of a typical electron beam irradiator is given in Figure 2.

Surface properties

The sugarcane bagasse collected from the sugar mill was ground and passed through a 100 µm sieve. All the samples were ground using a kitchen grinder and the ground particles were sieved using muslin cloth (100 µm approx.) and dried. On exposure to a defined dose, these samples were characterized using analytical techniques, such as SEM, XRD, FTIR, TGA-DTA and particle size analysis.

Scanning electron microscopic (SEM) studies

A scanning electron microscopic study was performed to analyze micro-structural changes that occurred on the surfaces of irradiated bagasse samples. The changes in surface morphology, phase distribution and microstructure of the polymer matrix were observed using SEM images. Prior to scanning, the samples were dried using a vacuum drying oven at 45 °C, after which they were mounted on sample holders and coated with gold. SEM images of the samples were recorded using an SEM Instrument, Korea, (Model no. SNE-4500 M), attached with a secondary electron detector, at a voltage of 10 kV.

Crystallinity measurement by X-ray diffraction (XRD) studies

The X-ray diffraction technique is one of the most important tools for characterization and determination of the crystallographic structure of a material. The X-ray diffraction patterns were recorded using X-ray Cu-Kα radiation at 40 kV and 40 mA, with the start angle of 5°, stop angle of 60°, with an Ultima-IV XRD diffractometer (Rigaku, Scintillation Tokyo, Japan). The ground bagasse samples (30-40 mg) were mounted on the holder and a proportional counter detector was set to collect data at $1°min^{-1}$, with an increment of 0.02° for 2Θ (theta) values. The scan speed was 6 °C/min.

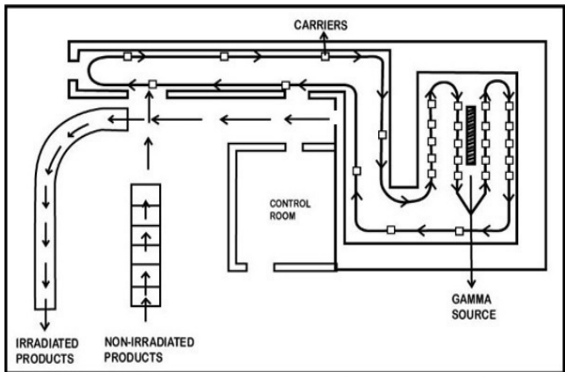

Figure 1: Schematic diagram of gamma irradiation unit using Co^{60}

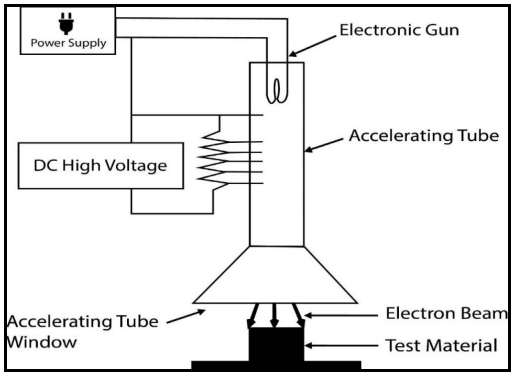

Figure 2: Schematic diagram of an electron beam irradiator

Particle size distribution

The particle size analysis was carried out in the dry mode, using a Microtrac S3500, USA, based on the laser diffraction technique, in the range between 0.2 µm to 2800 µm. A Turbotrac attachment (dry method using compressed air) was used to analyze the particle size of bagasse. The laser diffraction method evaluates the particle size distribution by measuring the angular variation in intensity of light scattered as a laser beam passes through a dispersed particulate sample. The values are represented in percentile. The sieves were stacked within the grooves on the bottom, with the sieve size increasing from top to bottom in the range of 1400-30 microns. The diameter was designated according to their distribution through each specific sieve number. D50 is also called as the median particle diameter or median particle size, *i.e.* if for a powder sample with D50 = 5 µm, it means 50% particles are larger than 5 µm and 50% particles are smaller than 5 µm.

Thermogravimetric/derivative thermogravimetric analysis (TGA/DTA)

The change in the thermal behaviour of sugarcane bagasse was studied using TGA/DTA (Model Perkin Elmer 4000, USA). The experiments were performed under continuous flow of N_2 gas with high purity. The experiments were carried out in the dynamic mode from ambient temperature to 1000 °C, with a heating rate of 20 °C per min. The mass of the sample used was between 6 to 15 mg. The weight loss of the samples against the rise in temperature was plotted to record degradation behaviour of non-irradiated and irradiated sugarcane bagasse samples.

Statistical analysis

The analysis of chemical constituents (cellulose, hemicelluloses, lignin and ash) was carried out in triplicate and the results were calculated as standard deviation. The mean values are presented in the graph. The error bars shown in the figure represent the standard error for triplicate evaluation. The data obtained were analyzed using 1-way ANOVA, and a p-value less than 0.05 was considered to be significant when compared to 0 kGy radiation dose.[37]

RESULTS AND DISCUSSION

Physical appearance

The changes in physical appearance of bagasse samples irradiated with gamma and e-beam were recorded. No appreciable change in the original off-white colour and hard texture of bagasse was observed up to 100 kGy exposure. However, the bagasse treatment at 500 kGy dose changed the colour of the samples to yellow, while the texture remained the same, in the case of both gamma and e-beam irradiation. On further exposure to a higher dose – of 1000 kGy, the bagasse became fluffy in texture and dark yellow in appearance (Table 1 and Fig. 3).

Chemical composition of sugarcane bagasse

Moisture content

With regard to the moisture content, it was remarked that longer exposure of bagasse to 2.5 kGy irradiation led to the conversion of a part of the energy into heat, resulting in the loss of moisture in the bagasse sample from 10 to 8% (Fig. 3). The higher rate of exposure of 2.5 kGy/second, in the case of e-beam exposure, was found to be a cold process, which did not affect the moisture content much. This might be explained by the shorter exposure time for imparting the same level of dose.

Bulk density

Upon increasing gamma dose exposure from 100 kGy to 1000 kGy, the bulk density of bagasse was found to increase from ~83 kg/m^3 to ~99 kg/m^3. The change in bulk density was found to be more prominent in the case of e-beam exposure; where it changed from to 83 kg/m^3 to 186 kg/m^3 at 1000 kGy.

The cellulose, hemicelluloses, lignin and ash content did not show any appreciable change up to the exposure dose of 100 kGy, in the case of both gamma and e-beam radiation (Table 2). This clearly indicates the disintegration of lignocelluloses is least up to the level of 100 kGy exposure. The cellulose content in the gamma irradiated bagasse decreased from 48 to 39% and 36% on exposure to 500 and 1000 kGy, respectively. Similarly, a major decrease of the cellulose content was observed after e-beam radiation exposure, which may be attributed to molecular disruption of the lignocellulosic matrix. There is a decrease in the hemicellulose content from 32 to 19.8% at 500 kGy, 16.2% at 1000 kGy exposure to gamma radiation. It may be attributed to the easy hydrolysability of the irradiated cellulose in equal terms of hemicelluloses. The lignin and ash contents do not show any changes upon radiation exposure (Table 2).

Table 1
Changes in physical appearance of bagasse after gamma and electron beam radiation exposure

Dose (kGy)	Physical appearance	
	Gamma	Electron beam
Control	Off-white and hard	Off-white and hard
100	Off-white and hard	Off-white and hard
500	Yellow and hard	Yellow and hard
1000	Dark yellow and fluffy	Yellow and fluffy

Figure 3: Physical appearance of irradiated and non-irradiated sugarcane bagasse

Table 2
Physico-chemical properties of irradiated and non-irradiated bagasse

Dose (kGy)	Bulk density (kg/m^3)	Moisture (%)	Cellulose (%)	Hemicelluloses (%)	Lignin (%)	Ash (%)
Control	83	10	48	31	19	4
Dose rate: 2.5 kGy/h (using γ-radiation)						
50	76	8.6	50	24.7	17.3	2.1
100	78	8.2	45	21.5	19.5	1.7
500	86	8.5	39	19.8	19.6	2.2
1000	99	8.7	36	16.2	19.4	2.4
Dose rate: 2.5 kGy/s (using e-beam)						
50	95	9.5	44	28	26.2	2.2
100	101	11.3	45	22	17.0	2.9
500	125	10.9	45	34	19.0	2.3
1000	186	11.1	16	39	23.1	1.9

Crystallinity study by XRD analysis

The relative crystallinity index of bagasse was calculated from the intensities of amorphous and crystalline regions measured at 2Ø (theta) = 18° and 2Ø (theta) = 22-22.5°, and their XRD patterns are given in Figure 5. The crystallinity index of cellulose showed a linear decreasing trend with an increasing dose of gamma radiation.[35]

The pattern of bagasse shows two humps, corresponding to amorphous cellulose at 16-20° and crystalline cellulose at around 20-25°; at higher radiation doses (500 and 1000 kGy), it indicates that the structure of crystalline cellulose was disrupted when bagasse is irradiated.

In the e-beam irradiated bagasse, there is only a slight decrease in the crystallinity index at 1000 kGy. In contrast, gamma irradiation of bagasse at 500 and 1000 kGy radiation dose led to a significant decrease in the crystallinity index (Fig. 4). In Figure 5b, two humps of crystalline and amorphous cellulose can be seen at 1000 kGy irradiation dose, which clearly suggests that e-beam does not have any significant effect on crystallinity at different doses of irradiation.

Scanning electron microscopy (SEM)

The morphology of sugarcane bagasse was studied using a scanning electron microscope. SEM revealed structural changes in bagasse upon gamma and e-beam irradiation. The SEM

images of the non-irradiated sugarcane bagasse are shown in Figure 6.

The morphology observation of sugarcane bagasse reveals a heterogeneous surface. Nice spider type networks of microfibril celluloses, rod-shaped microcrystalline cellulose and leafy type shape of lignocellulose can be observed in the SEM micrographs. These shapes are indicative of the complex structure and heterogeneity in the components of biomass. The creation of pores in the leafy structure of the bagasse indicates destruction of the lignocellulosic matrix upon gamma and e-beam irradiation Figure 7 (A, B, C, D). In the lignocellulosic structure, cellulose microfibrils are protectively surrounded by hemicelluloses and lignin. Therefore, both gamma and e-beam irradiation disrupted the hemicelluloses and lignin, creating pores that opened up the lignocellulosic matrix. After irradiation at 1000 kGy, cleavage of the fibers in the external layers was observed with an increase in surface roughness and porosity.

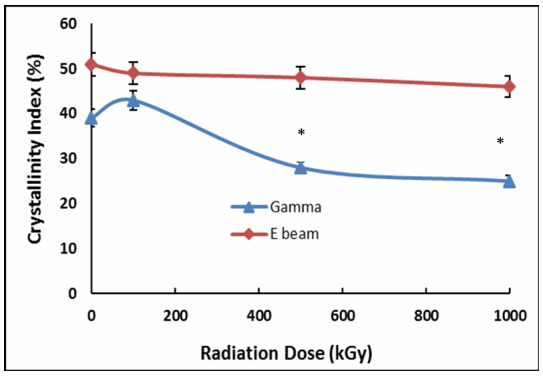

Figure 4: Crystallinity index of sugarcane bagasse (data represent mean ± SE of 3 values; *p < 0.05, significant when compared to zero kilo Gray radiation dose)

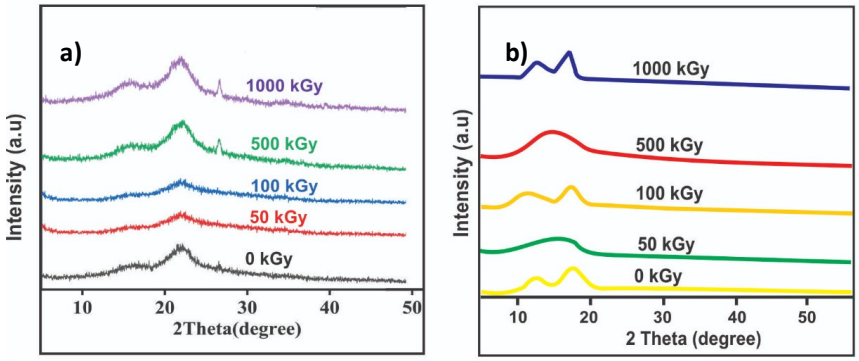

Figure 5: X-ray diffraction patterns of a) gamma and b) electron beam irradiated bagasse at different doses

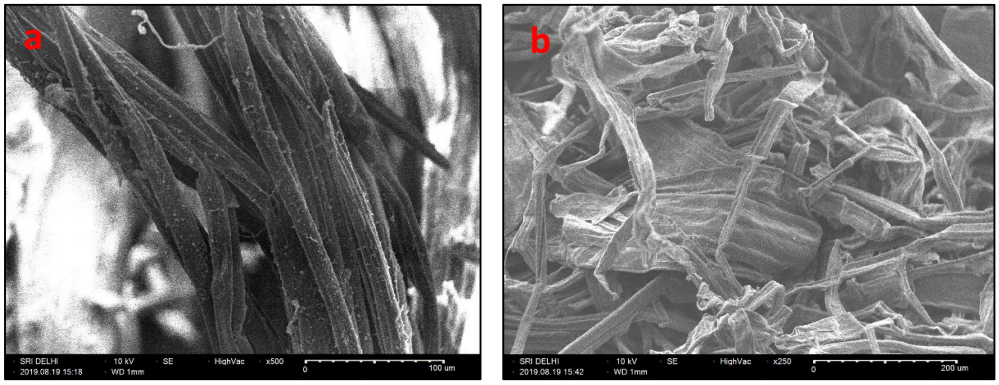

Figure 6: SEM images of non-irradiated sugarcane bagasse (a, b) at (500 and 250x) magnification

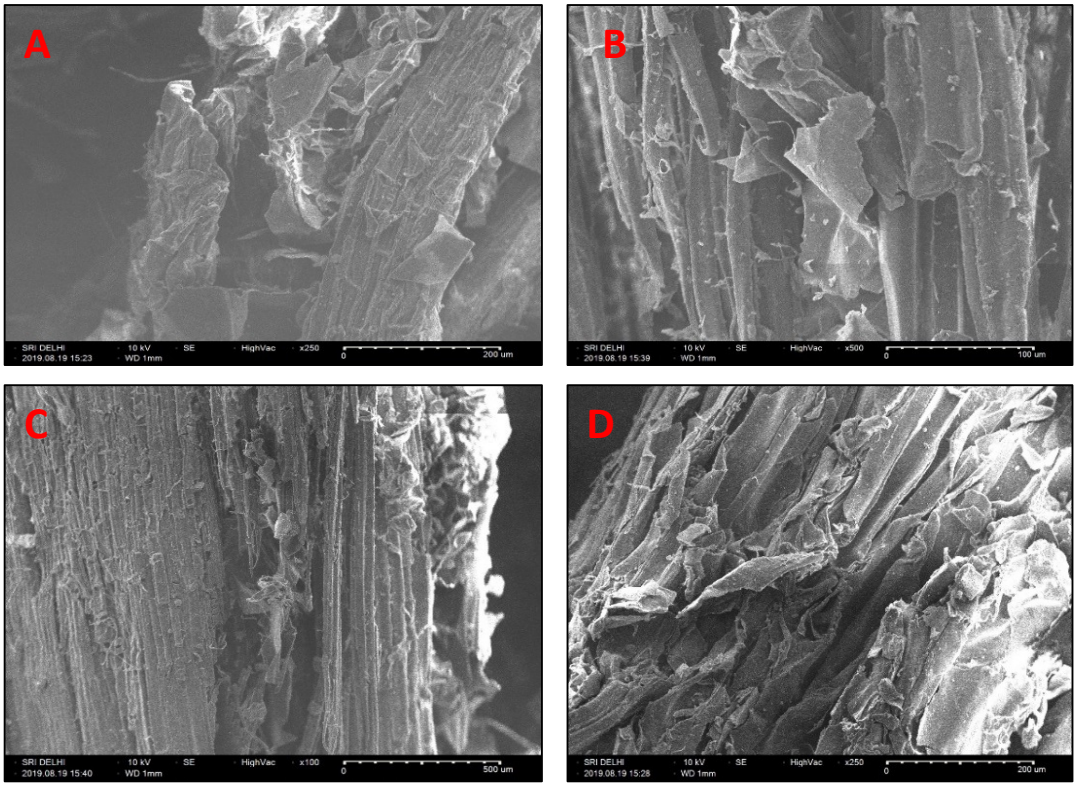

Figure 7: SEM images of irradiated sugarcane bagasse showing distorted structures of bagasse fibre due to the effect of gamma (A and B) and e-beam (C and D) irradiation (A and C – 250x, B and D – 500x)

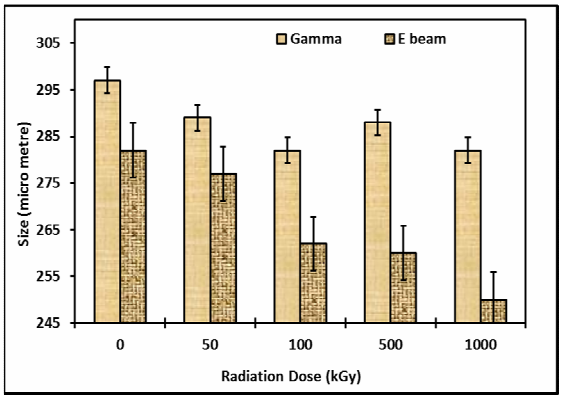

Figure 8: Particle size of gamma and e-beam irradiated bagasse

Particle size distribution

To evaluate the physical changes occurring in bagasse upon irradiation, the particle size distribution of the raw bagasse and the irradiated materials was analyzed. The particle size distribution plays an important role in controlling the surface area that will be available for chemical exposure of the biomass during hydrolysis. A comparison in particle size values (D50) of gamma and e-beam irradiated sugarcane bagasse is shown in Figure 8. Apparently, when the bagasse is irradiated, fragmentation phenomena occur. There is a decrease in particle size with increasing radiation dose. The effect is more prominent in electron beam irradiated bagasse, compared to gamma irradiated bagasse. This reveals that the particles of the lignocellulosic structure of

bagasse are disintegrated by high energy electrons. When the dose rate is increased, the particle size of bagasse decreases as well (Fig. 8). Moreover, there is a drop in the values of gamma irradiated bagasse as the dose rate is increased to 1000 kGy, which signifies less fragmentation has occurred, as compared with the e-beam irradiated samples.

Thermogravimetric (TGA/DTA) analysis

To figure out the effects of radiation on the thermal behaviour of bagasse, derivatives of thermogravimetric analyses (TGA-DTA) are shown in Figures 9 and 10. The DTA curve at 0 kGy shows initial degradation peaks at 300 and 339 °C, but at 1000 kGy gamma exposure, there is a change in the degradation behaviour of the cellulosic portion of bagasse, where the peak was observed at 295 and 326 °C. No specific change appeared at 100 kGy radiation dose, as the peaks can be seen at around 301 and 340 °C. It is inferred that this double peak distribution is due to thermal degradation of cellulose and hemicelluloses present in bagasse (Fig. 9).

Further, in e-beam irradiated bagasse, there is a slight shift in degradation temperature. The DTG peak shifts to 318 and 365 °C at 100 kGy dose, and to 308 and 330 for 1000 kGy, which also indicates thermal degradation of the biomass polymer. However, gamma exposure, due to high penetration effect, led to changes in the degradation temperatures. In gamma-irradiated bagasse, the initial degradation appears at around 295 °C, whereas in the case of e-beam, it is at 308 °C. Hence, it shows that gamma and e-beam pretreatment have pronounced effects on the degradation of polymeric chains.

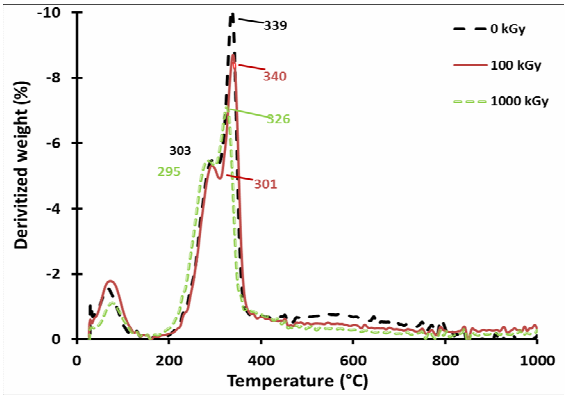

Figure 9: DTA curves of gamma irradiated bagasse

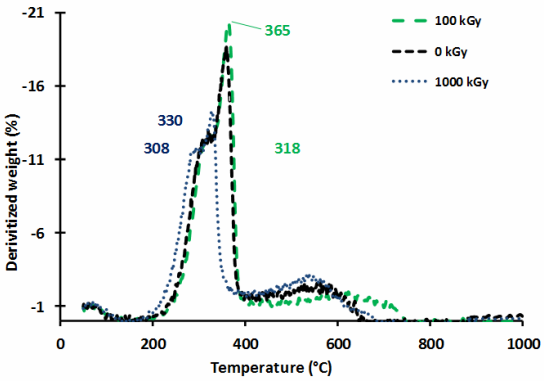

Figure 10: DTA curves of e-beam irradiated bagasse

Table 3
Summarized results of gamma and e-beam irradiated bagasse

Parameter	Result	
	Gamma	Electron beam
Cellulose content	36	16
Hemicellulose content	16.2	39
Moisture	8.7	11.1
Bulk density	99	186
Lignin and ash	2.4	1.9
Crystallinity	Crystallinity index	Crystallinity index
SEM images	Disruption in the matrix due to irradiation	Disruption in the matrix due to irradiation
Particle size	Less fragmentation as compared with e-beam	Fragmentation occurs
DTA	Double peak distribution due to thermal degradation	Slight shift in degradation temperature

CONCLUSION

From this study, it is concluded that the irradiation pretreatment of sugarcane bagasse has a considerable impact on the degradation of cellulose and hemicellulose chains. A comparative study was carried out on to find out the effects of gamma and e-beam radiation on sugarcane bagasse. SEM images show partially

distorted surface caused by irradiation. The particle size of bagasse fibers undergoing irradiation treatment were significantly affected by fragmentation. The thermal degradation is also evident in DTA analysis, which revealed changes in the degradation temperature.

The chemical and structural changes in sugarcane bagasse mainly arise due to high energy radiation treatments. The results also suggest that gamma irradiation was found to be the more effective of the two sources, in terms of the chemical and morphological changes induced. This study is important as a future perspective, since irradiation treatment can be used in combination with other pretreatment processes, in order to achieve efficient hydrolysis of cellulose and hemicelluloses, avoiding the loss of reducing sugars. The simplicity of this process and the effects of chain cleavage are its major advantages.

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