# Control of Resin Release from Particleboards by Gamma Irradiation. I. Thermal Decomposition Behavior and Structure Morphology

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*Received 30 September 2000; accepted 3 March 2001*

**ABSTRACT:** The release or emission of resins from pressed particleboards, modified through gamma radiation, was characterized in terms of the thermal decomposition (TGA) and structure morphology (SEM). Particleboards, based on some farm residues and some polymers as adhesives, were first made by thermal compression in a hot press at 120°C and subsequently exposed to various doses of gamma irradiation. In general, gamma irradiation improves the thermal stability of the particleboards regardless of the type of the farm residues or the type of adhesive. Meanwhile, the thermal stability was found to increase with increasing irradiation dose as shown by the percentage loss in weight at different decomposition temperatures and the temperatures of the maximum values of the rate of reaction. The results showed that the particleboards based on cotton or flax stalks and polystyrene (PS) displayed higher thermal stability than did those based on the epoxy resin (E150). The particleboards based on wood sawdust and the E150 resin showed higher thermal stability than did those based on PS. SEM observations of the fracture surfaces of the different particleboards give further support to the improvement in the thermal properties after exposure to gamma radiation. In this regard, the pores and distances between the base material were coated with radiation-crosslinked or -grafted E150 resin, particularly in the case of wood sawdust boards. © 2001 John Wiley & Sons, Inc. J Appl Polym Sci 82: 2869–2881, 2001

**Key words:** particleboards; gamma irradiation; thermal stability; morphology

## INTRODUCTION

There has been increasing interest in recent years to prepare particleboards from farm residues[1–5] or wood polymer composites (WPC) based on recycled products and acrylate polymers initiated by either chemical or radiation processing.[6–10] Low-density particleboards based on paddy straw and coconut pitch using urea formaldehyde (UF) as a binding material are suitable for insulation purposes.[11] The thermal conductivity and flammability of WPC based on tropical woods, prepared by polymerizing monomers *in situ* in oven-dried woods by gamma radiation, were investigated.[12] It was shown that WPC based on polyacrylonitrile displayed the greatest reduction in thermal conductivity and those based on polystyrene/polyacrylonitrile exhibited the least.

Few reports were concerned with using scanning electron microscopy (SEM) to examine the structural morphology of either particleboard products or wood fiber composites to illustrate the

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Journal of Applied Polymer Science, Vol. 82, 2869–2881 (2001)

bending models. SEM was utilized to observe the microstructure and fracture surfaces of tensile bending and internal bonding of fiber/recycled tire rubber composites.[13] The results showed that excellent bonding was formed between fibers, and good bonding was also observed between fiber and rubber in the SEM micrographs.

Although particleboards based on UF resins represent most of the produced materials in industrial scale, they show lower stability under humid and warm conditions. This unstability is attributed to hydrolysis and hydrolytic degradation and stress.[14] This problem indicates that thermally crosslinked UF does not become part of the structure during the curing process, which facilitates its release or emission on usage. The improvement in the stability and durability of pressed products would increase their application and reduce the toxicity of these resins.

A big effort is being done to minimize this release,[15–17] for example, by lowering the molar ratio of formaldehyde to urea in the adhesive by addition of scavengers after treatment with ammonia or urea and also by other modifications of the resin and panel manufacturing process.[18] The durability and stability of UF-bonded wood products were improved by decreasing the internal stress developed during resin cure and by improving the ability of the cured system to withstand cyclic stresses.[14] UF was modified by incorporating urea-terminated di- and trifunctional flexible amines or by curing the resins with hydrochloride derivatives of some of these amines in place of ammonium chloride.

Thus, it was aimed in this article to present a balanced view to overcome this problem by introducing radiation curing and/or grafting after thermal treatment. This approach will eventually depend on how sensitive the chosen resins to fill the voids of particleboard are to radiation-initiated crosslinking. The waste materials used in this study were cotton stalks, flax stalks, and wood sawdust, while the adhesives were polystyrene and some commercial epoxy resin. The effect of gamma irradiation was illustrated in terms of the changes in the thermal stability using thermogravimetric analysis (TGA) and structure morphology using SEM.

## EXPERIMENTAL

### Materials

The waste materials used throughout this work for the preparation of the particleboards were cotton stalks, flax stalks, and wood sawdust. The cotton and flax stalks were collected from farm residue. However, before use, they were cleaned of dust and fiber residue and crushed into small pieces. The wood sawdust was a by-product produced in a carpenter workshop and it is a mixture of different kinds of woods usually used in the furniture industry. The polymers used as adhesives for the particleboards were polystyrene (PS) and kemapoxy 150 (E150). Kemapoxy 150 is a commercial product based on specifically modified epoxy resins purchased from the Chemicals for Modern Building Co. (Cairo, Egypt). It is a two-component product and solvent free, and it is used for repairing mortar and floor toppings. The PS used was a pure grade (purchased from Aldrich Chemicals Co., Milwaukee, WI) in the form of pellets and has an average molecular weight of 280,000.

### Preparation of Particleboards

The crushed cotton or flax stalks and wood sawdust were first conditioned to remove the contained moisture by placing them in an oven at 105°C for 2 h. The conditioned materials were thoroughly mixed with various ratios of the polymeric adhesives under investigation dissolved in the appropriate solvent. The mixture was then compressed in a hot press at 120°C for various lengths of time to form sheets of dimensions of 10 mm in thickness and 100 mm in width and length. The particleboard sheets were prepared under a constant pressure of 12 tons/m$^2$.

### Gamma Irradiation

Irradiation to the required doses was carried out with a $^{60}$Co gamma source (made in India) at the National Center for Radiation Research and Technology (Cairo, Egypt). The particleboards were exposed to gamma irradiation in air at a dose rate of 0.40 Mrad/h.

### Thermogravimetric Analysis

The thermal decomposition behavior of the different particleboards was investigated by TGA using a TG-50 instrument from Shimadzu (Japan) at a heating rate of 10°C/min. The data from the TGA curves were used to determine the percentage weight loss at different decomposition temperatures and the temperatures of the maximum values of the rate of reaction.

### SEM Analysis

The morphology of the fracture surfaces of the different particleboards was examined by SEM.

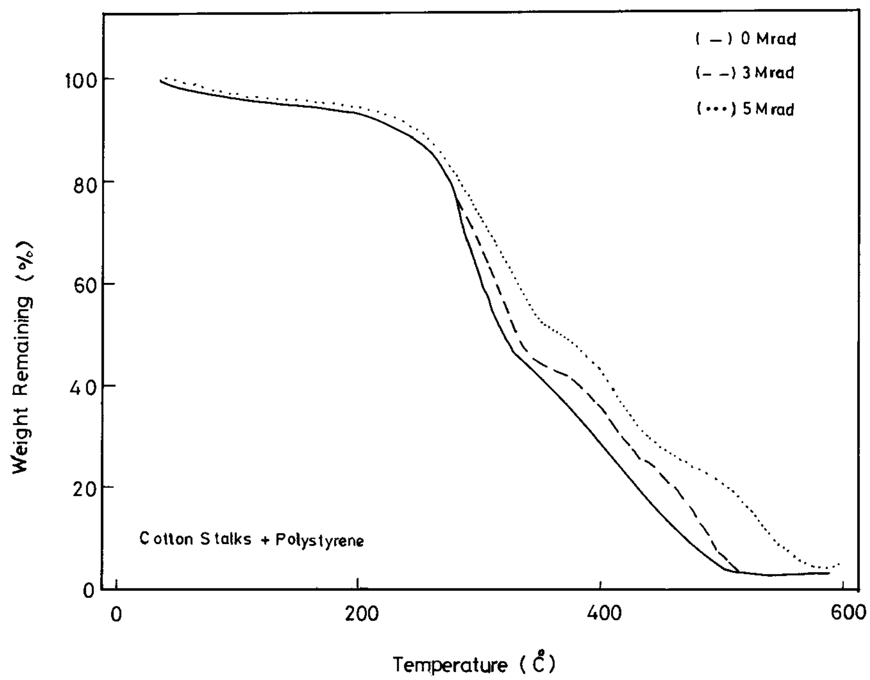


**Figure 1** TGA thermograms of particleboards based on cotton stalks and PS (20 wt %) as an adhesive initially made by thermal pressing at 120°C for 20 min and exposed to various doses of gamma radiation.

The SEM micrographs were taken with a JSM-5400 (JEOL, Japan). A sputter coater was used to precoat conductive gold onto the fracture surface before observing the microstructure at 20 and 30 kV.

## RESULTS AND DISCUSSION

In the present work, the effect of gamma irradiation on the fixation of different polymers (used as adhesives) into the bulk of particleboards prepared by thermal curing was investigated. The fixation of resins by gamma radiation is expected to minimize the release or emission during their lifetime. The enhancement in thermal stability, static bending properties, water absorption and thickness swelling, and structure morphology could be taken as a measure for this fixation. In this part, the thermal stability and structure morphology will be presented.

### Thermal Decomposition Behavior of Particleboards Based on Cotton Stalks

Different particleboards prepared by thermal curing before and after they had been exposed to doses of 3 and 5 Mrad gamma radiation were subjected to thermogravimetric analysis using a method based on the rate of conversion of the material at a single heating rate. Figures 1 and 2 show the primary TGA thermograms of particleboards based on cotton stalks as a base and PS or epoxy 150 (E150) as adhesives before and after they had been exposed to doses of 3 and 5 Mrad. The percentage weight loss at different decomposition temperatures for the same boards taken from the TGA thermograms is summarized in Tables I and II. Within the decomposition temperature range of 100–200°C, the nonirradiated or irradiated particleboards based on either PS or EI50 have the same thermal stability, in which an approximately equal loss in weight can be observed. However, in all cases, the irradiated boards are more stable than are the nonirradiated ones. By increasing the heating temperature from 200 to 300°C, the non-irradiated or irradiated particleboards based on PS seem to possess higher thermal stability than that of those based on EI50. Meanwhile, it can be seen that the percentage loss in weight of both boards was found to decrease with increasing irradiation dose. The nonirradiated particleboards based on E150 are thermally more stable than are those based on PS

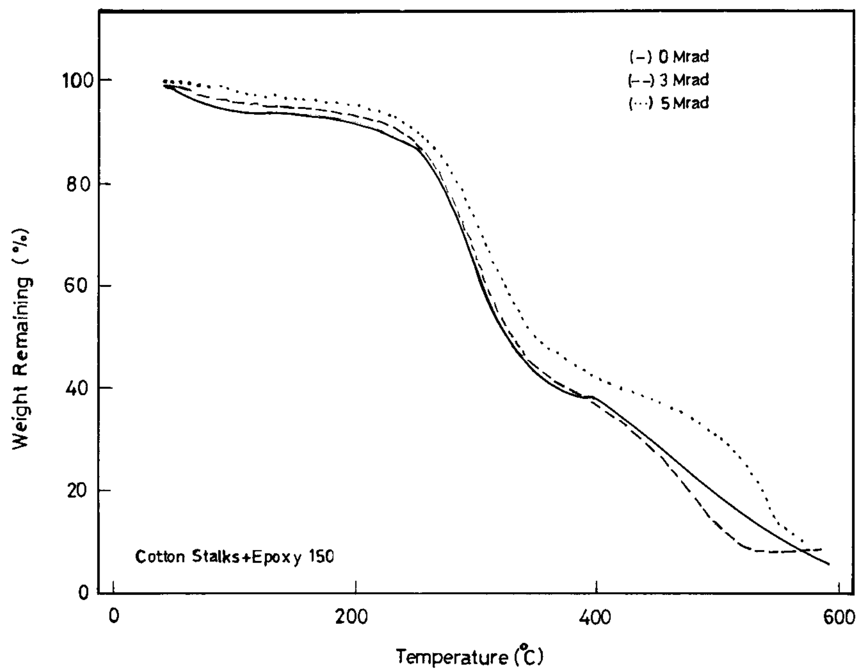


**Figure 2** TGA thermograms of particleboards based on cotton stalks and Epoxy 150 (20 wt %) as an adhesive initially made by thermal pressing at 120°C for 20 min and exposed to various doses of gamma radiation.

at 400 and 500°C. While the particleboards based on PS or E150 irradiated to a dose of 3 Mrad have approximately the same thermal stability, irradiated boards based on PS are thermally more stable than are those based on E150 at 5 Mrad.

As can be seen, the thermal stability of the particleboards is greatly dependent on the type of adhesive which binds the cotton stalks in the bulk of the boards. As reported in a previous work, the calculated average complete dissociation energy of PS is about 101.5 kcal/mol and that for acrylate polymers is about 99.1 kcal/mol.[19] It is to be noted that these calculations are based on the strength of the covalent bond between the atoms forming the polymer molecules. Thus, the relatively higher thermal stability obtained by experimental TGA ther-

**Table I Weight Loss (%) at Different Decomposition Temperatures for Particleboards Based on Different Waste Materials and PS (20 wt %) Exposed to Various Doses of Gamma Radiation**

| Type of Waste Material | Irradiation Dose (Mrad) | Weight Loss (%) | | | | |
|---|---|---|---|---|---|---|
| | | 100°C | 200°C | 300°C | 400°C | 500°C |
| Cotton stalks | Unirradiated | 5.46 | 9.66 | 38.59 | 68.36 | 95.91 |
| | 3 | 4.76 | 7.08 | 37.08 | 61.41 | 80.87 |
| | 5 | 3.69 | 6.71 | 31.90 | 55.69 | 69.74 |
| Flax stalks | Unirradiated | 3.13 | 6.25 | 32.50 | 67.50 | 86.88 |
| | 3 | 3.12 | 6.22 | 28.87 | 65.29 | 82.47 |
| | 5 | 3.10 | 6.21 | 32.50 | 65.04 | 76.55 |
| Wood sawdust | Unirradiated | 5.08 | 8.33 | 36.16 | 73.45 | 94.92 |
| | 3 | 4.89 | 7.34 | 29.89 | 71.05 | 94.96 |
| | 5 | 2.78 | 6.66 | 29.89 | 71.30 | 94.44 |

The particleboards were initially made by thermal pressing at 120°C for 20 min at a constant pressure of 12 ton/$m^2$.

**Table II Weight Loss (%) at Different Decomposition Temperatures for Particleboards Based on Different Waste Materials and Epoxy 150 (20 wt %) Exposed to Various Doses of Gamma Radiation**

| Type of Waste Material | Irradiation Dose (Mrad) | Weight Loss (%) | | | | |
|---|---|---|---|---|---|---|
| | | 100°C | 200°C | 300°C | 400°C | 500°C |
| Cotton stalks | Unirradiated | 6.37 | 9.16 | 44.22 | 64.32 | 88.44 |
| | 3 | 4.52 | 7.54 | 43.03 | 61.75 | 79.68 |
| | 5 | 3.96 | 6.93 | 37.62 | 59.90 | 73.76 |
| Flax stalks | Unirradiated | 4.30 | 7.03 | 37.34 | 68.65 | 90.13 |
| | 3 | 3.11 | 6.72 | 33.79 | 65.76 | 79.00 |
| | 5 | 3.53 | 6.22 | 31.52 | 65.22 | 78.00 |
| Wood sawdust | Unirradiated | 7.22 | 7.22 | 28.67 | 60.72 | 69.53 |
| | 3 | 6.48 | 6.48 | 28.33 | 61.43 | 73.33 |
| | 5 | 6.14 | 6.14 | 25.60 | 58.43 | 71.35 |

The preparation conditions of particleboards are the same as in Table I.

mograms of these materials can be explained on the basis of these theoretical calculations. Moreover, it was reported that PS is a radiation-resistant polymer compared to acrylate polymers.

The rate of conversion *dw/dt* or the derivative of the thermogravimetric analysis curve (DTGA) taken from the initial TGA is plotted against the decomposition temperature as shown in Figures 3 and 4. It should be noted that the numerical values on the rate of reaction axis were not shown because all curves start from the zero point. It can be seen that this type of curve displays similar trends; however, the temperature of the maximum value of the rate of reaction differs from one board to another as shown in Table III. Moreover, it can be observed that the rate of reaction for all particleboards, either before or after gamma irradiation, expresses three maxima with increasing temperature. The first maximum is probably due to the release of included moisture inside the board structure, while the second and third maxima are due to the decomposition of cellulose and the used adhesive. As shown in Table III, the second maximum was not affected by gamma irradiation; however, the temperature of the third maximum was affected upon exposure to 3 and 5 Mrad. It is clear that the temeperature of the third maximum indicates that the thermal stability of the boards based on PS is much higher than that of those based on EI50. Also, this temperature was found to increase with increasing irradiation dose. This finding may be explained on the basis that the PS polymer undergoes crosslinking upon gamma irradiation of particleboards.

The particleboards were cured at 120°C before exposure to gamma radiation because this temperature, according to the standard industrial manufacturing levels of particleboards, is not sufficient for complete thermal curing. For this reason, it was intended to expose the cured boards

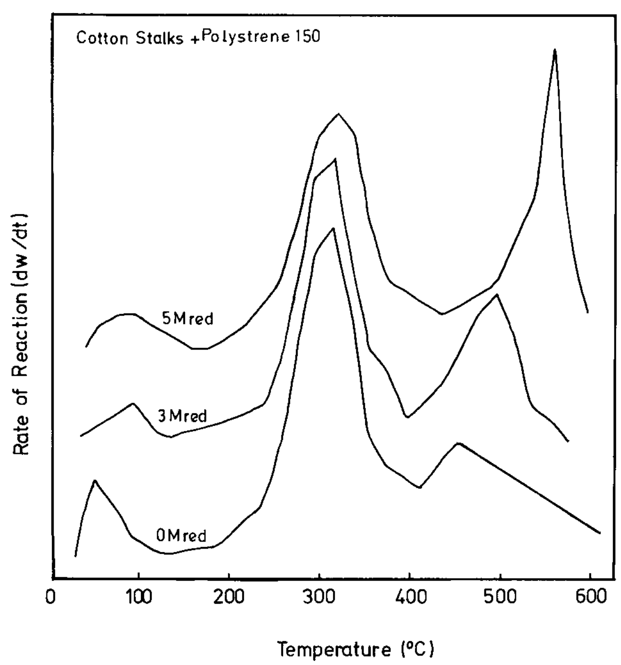


**Figure 3** Representative curves of the rate of reaction (*dw/dt*) against temperature for particleboards based on cotton stalks and PS (20 wt %) as an adhesive initially made by thermal pressing at 120°C for 20 min and exposed to various doses of gamma radiation.

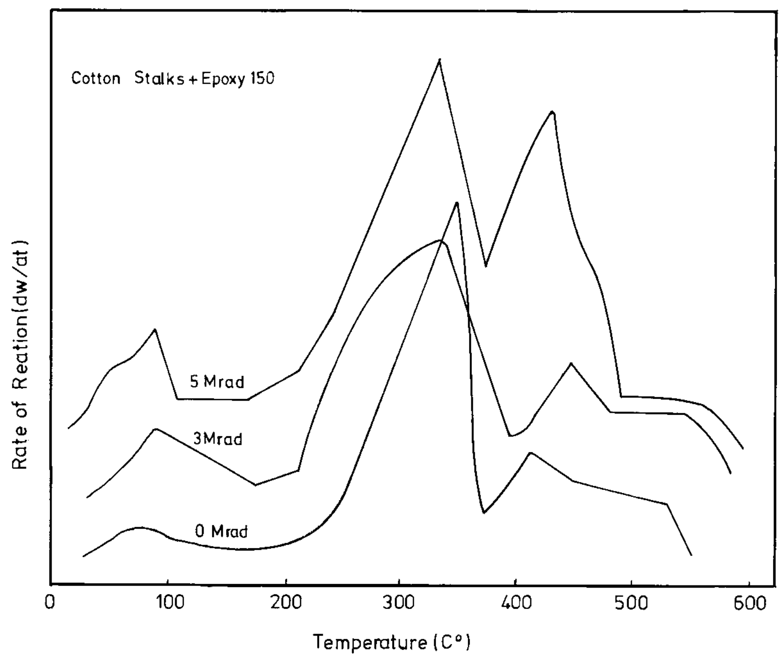


**Figure 4** Representative curves of the rate of reaction (*dw/dt*) against temperature for particleboards based on cotton stalks and Epoxy 150 (20 wt %) as an adhesive initially made by thermal pressing at 120°C for 20 min and exposed to various doses of gamma radiation.

directly to gamma radiation. Thus, the enhancement in the thermal stability of cotton stalks based on different polymeric adhesives may be explained as follows: The interaction of radiation with PS was reported to produce two types of radical intermediates.[20] The *G*(*X*) values are be-

**Table III Temperatures of the Maximum Value of the Rate of Reaction for Particleboards Based on Different Waste Materials and Different Adhesives (20 wt %) Exposed to Various Doses of Gamma Radiation**

| Type of Waste Material | Irradiation Dose (Mrad) | Temperatures of the Maximum Rate of Reaction (°C) | | | | | |
|---|---|---|---|---|---|---|---|
| | | PS | | | Epoxy 150 | | |
| | | $1^{st}$ Stage | $2^{nd}$ Stage | $3^{rd}$ Stage | $1^{st}$ Stage | $2^{nd}$ Stage | $3^{rd}$ Stage |
| Cotton stalks | Unirradiated | 52 | 312 | 452 | 70 | 350 | 410 |
| | 3 | 74–94 | 312 | 493 | 92 | 338 | 441 |
| | 5 | 93 | 312 | 554 | 91 | 331 | 431 |
| Flax stalks | Unirradiated | 53 | 313 | 413 | 70 | 310 | 490 |
| | 3 | 73 | 333, 433 | 533 | 70 | 330 | 550 |
| | 5 | 52–72 | 350 | 600 | 70 | 330 | 570 |
| Wood sawdust | Unirradiated | 75 | 335 | 473 | 75 | 355 | — |
| | 3 | 75 | 353 | 473 | 73–93 | 333 | 553 |
| | 5 | 75 | 350 | 510 | 84 | 344 | 584 |

The preparation conditions of particleboards are the same as in Table I.

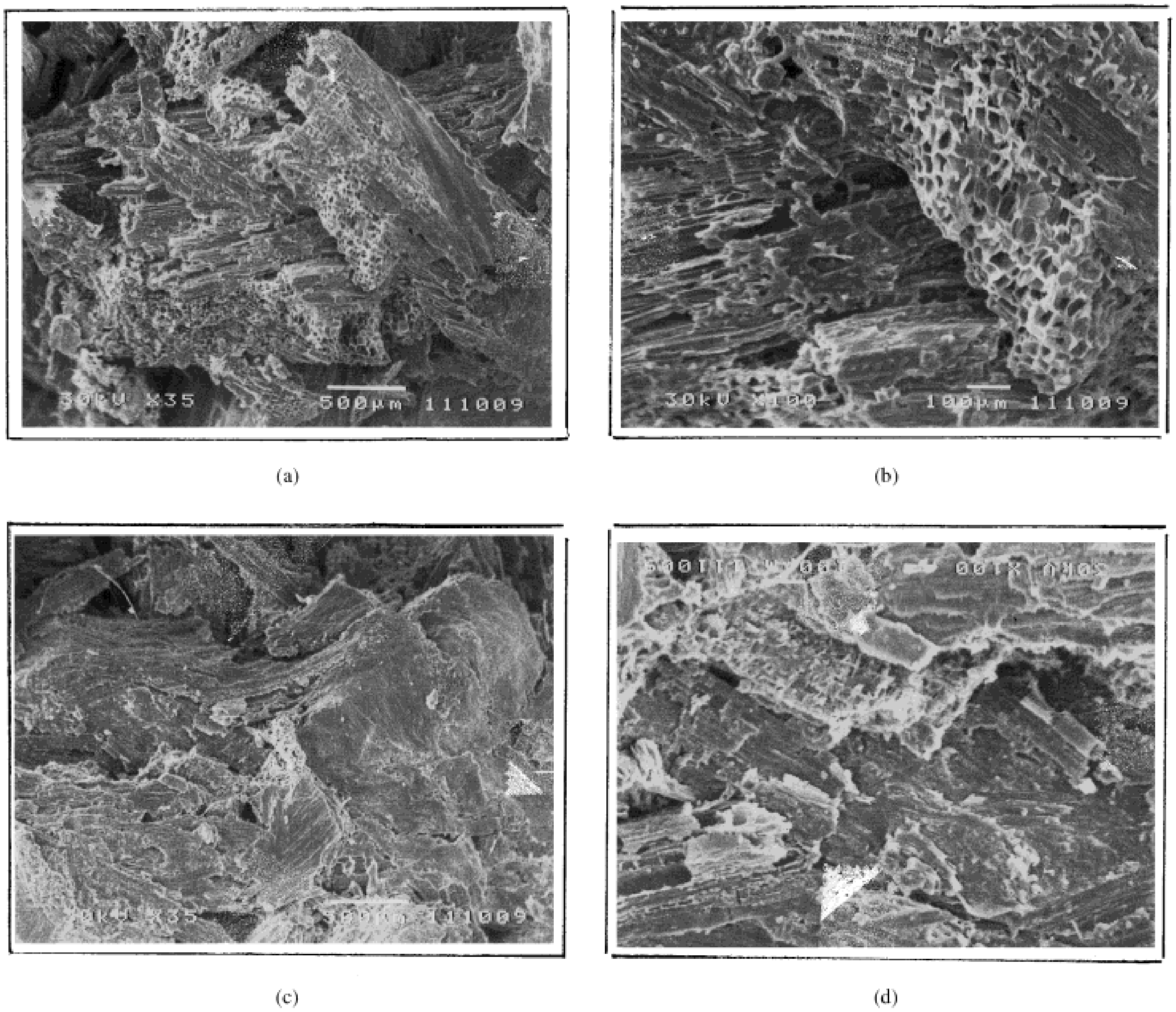


**Figure 5** SEM micrographs at different magnifications of particleboards based on cotton stalks and 20 wt % EI50: (A,B) unirradiated; (C,D) irradiated to 5 Mrad.

tween 0.02 and 0.54, while *G*(*S*) was reported to be below 0.02. Thus, crosslinking of PS inside the matrix of the boards is possible. Also, it is possible that PS or acrylate resins (E150) are grafted onto the cellulose molecules of cotton stalks, which, in turn, become part of the boards.

## Particleboards Based on Flax Stalks

The percentage loss in weight for the thermal decomposition behavior of particleboards based on flax stalks and different adhesives before and after exposure to gamma irradiation is shown in Tables I and II. For particleboard based on PS, it can be seen that there is no difference in weight loss percent between the nonirradiated and irradiated boards up to a heating temperature of 200°C. On the other hand, the weight loss percent of the boards based on EI50 was found to decrease after exposure to gamma irradiation. By further heating from 200 to 500°C, the nonirradiated or irradiated boards based on PS are thermally more stable than are those based on EI50.

The temperatures of the maximum values of the rate of reaction (*dw*/*dt*) for flax stalks boards based on PS and E150 before and after they had been exposed to 3 and 5 Mrad as taken from the corresponding *dw*/*dt*–temperature curves (not shown) are shown in Table III. It is clear that the unirradiated flax board based on EI50 with a higher third $T_{max}$ (490°C) is more stable against thermal decomposition than is that based on PS in which $T_{max}$ equals 413°C as shown in Table III. This is may be attributed to the greater accessibility of flax stalks for acrylate resins than for PS. However, flax board based on PS irradiated to 5

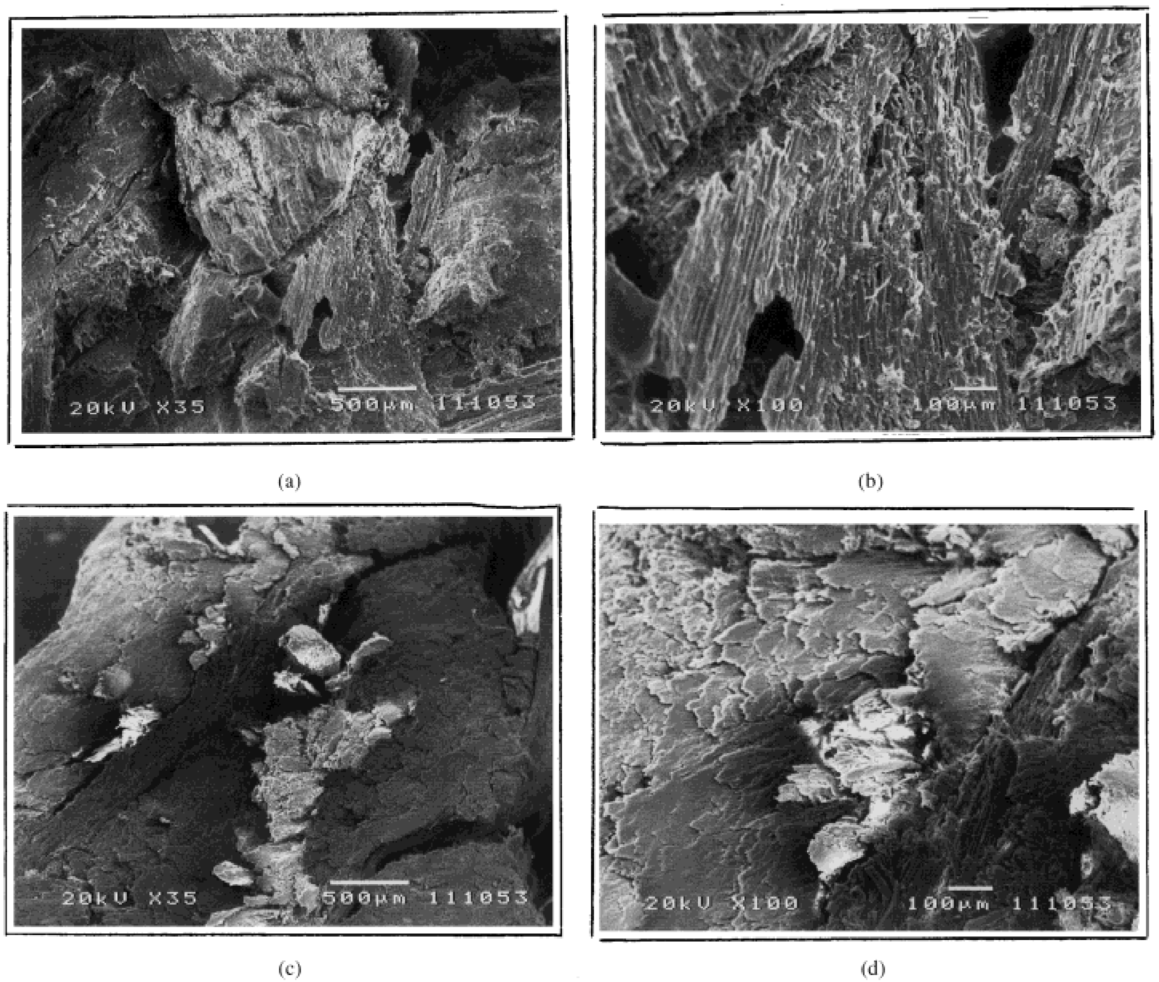


**Figure 6** SEM micrographs at different magnifications of particleboards based on cotton stalks and 20 wt % PS: (A,B) unirradiated; (C,D) irradiated to 5 Mrad.

Mrad displayed a higher thermal stability with a $T_{max}$ of 600°C than that based on EI50 with a $T_{max}$ of 570°C.

## Particleboards Based on Wood Sawdust

Wood sawdust as a waste material is usually composed of different kinds of woods. Also, unlike cotton and flax stalks, the size of the particles is relatively very small. Thus, the mixture of sawdust and the dissolved adhesives will be more homogeneous than in the case of other boards. In a similar manner, the TGA thermograms and the rate of the reaction–temperature curves were carried out for wood sawdust particleboards based on PS and EI50 as adhesives (not shown). The percentage weight loss percent at different decomposition temperatures and the temperatures of the maximum values of the rate of reaction are summarized in Tables I–III. At the beginning of thermal heating, the board based on PS either before or after irradiation starts to decompose with less percentage weight loss than that based on the EI50 resin. Within the decomposition temperature range 200–500°C, the nonirradiated or irradiated boards based on EI50 are thermally more stable than are these boards based on PS. The differences in the temperatures of maximum values of the rate of reaction give further support to the respective thermal stability for the boards based on both types of adhesives as shown in Table III.

On the basis of the results of the thermal decomposition behavior of the different particleboards, two points may be addressed: (1) The thermal stability (on the basis of weight loss percent) of the nonirradiated boards based on PS overall the studied decomposition temperature

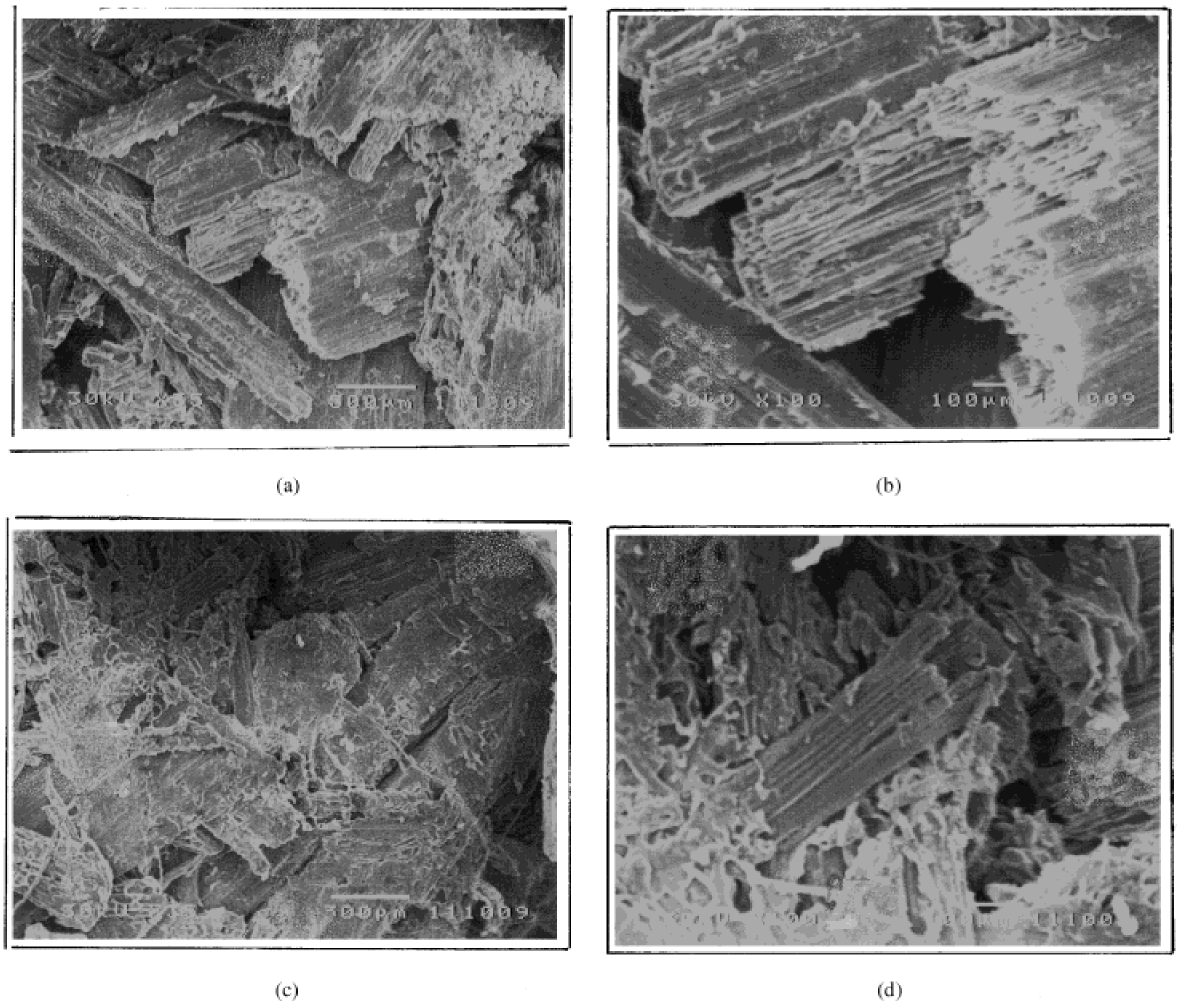

**Figure 7** SEM micrographs at different magnifications of particleboards based on flax stalks and 20 wt % EI50: (A,B) unirradiated; (C,D) irradiated to 5 Mrad.

ranges can be arranged according to the type of waste material as follows: flax stalks > wood sawdust > cotton stalks. In the case of E150 as a polymer adhesive, the thermal stability of the nonirradiated particleboards can be arranged as follows: flax stalks > wood sawdust > cotton stalks (up to 200°C) and wood sawdust > flax stalks > cotton stalks (up to 500°C). Within the higher decomposition temperature range of 300–500°C, the nonirradiated wood sawdust/E150 boards displayed the highest thermal stability overall the other particleboards. (2) In general, irradiated particleboards are thermally more stable than are the nonirradiated ones regardless of the type of waste material or polymer adhesive. The highest improvement in thermal stability was observed in the case of wood sawdust/E150 and cotton stalks/PS boards irradiated to 5 Mrad, particularly within the decomposition temperature range 300–500°C.

## Structure Morphology

SEM was used to study the bonding between the waste material and the adhesives EI50 and PS in the microstructure of the particleboards, before and after they had been exposed to a dose of 5 Mrad gamma radiation. Figure 5 shows SEM micrographs of particleboards based on cotton stalks and the EI50 resin before and after gamma irradiation. For unirradiated particleboards [Fig. 5(A,B)], the pores of the cotton stalks are partially filled with the resin EI50. Also, it can be seen that there is no sufficient interfacial bonding between the cotton stalks and the resin. These observations may indicate that there is an excess of un-

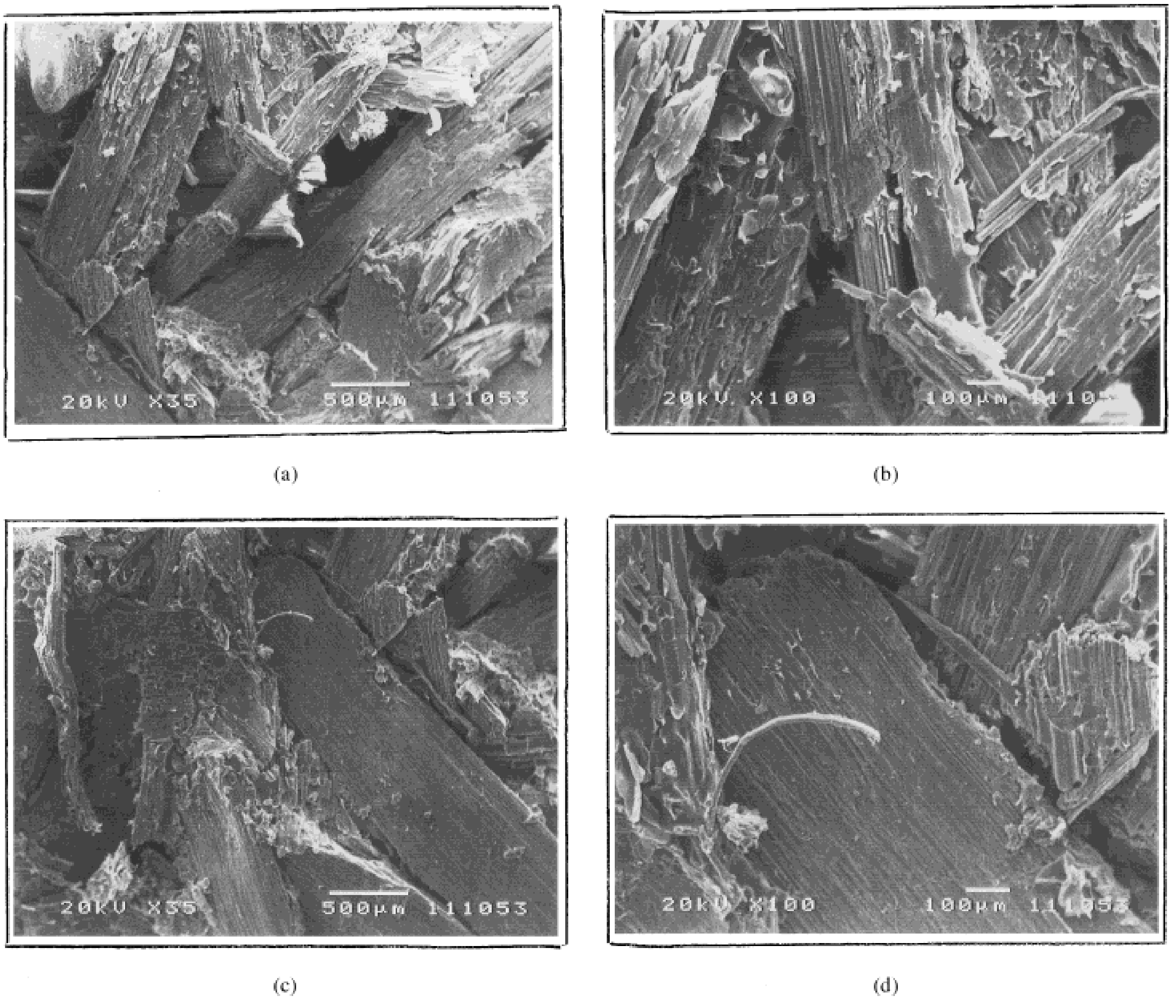


**Figure 8** SEM micrographs at different magnifications of particleboards based on flax stalks and 20 wt % PS: (A,B) unirradiated; (C,D) irradiated to 5 Mrad.

cured resin inside the pores of the cotton stalks. This is because cotton stalks, from the point of view of morphology, are long fibrous material and the resin was applied in the liquid form. Moreover, it can be observed that cotton stalks are aligned horizontally along the fracture surfaces of particleboards. A different microstructure feature can be seen for the same particleboards after exposure to gamma radiation as shown in Figure 5(C,D). The pores of the cotton stalks are completely covered and filled with the resin E150, suggesting the formation of additional crosslinking of the resin induced by gamma irradiation. Moreover, a strong interfacial bonding was developed between the cotton stalks and the resin resembling the occurrence of graft polymerization. Figure 6 shows SEM micrographs at different magnifications of particleboards based on cotton stalks and PS as an adhesive before and after exposure to gamma irradiation. For the unirradiated boards, the spacing and cavities between the stalks are large in size compared to those in the case of E150 resin as shown in Figure 6(A,B). This indicates that the compatibility between the cotton stalks and PS is less than that with E150. However, the pores and cavities are completely covered and filled with the radiation-cured PS as shown in Figure 6(C,D). This structure may illustrate the higher thermal stability and, eventually, lower water absorption properties of cotton stalks/PS boards than those of cotton stalks/E150 boards.

As shown in Figure 7(A,B), unlike cotton stalks, unirradiated flax stalks are nonporours material constituting a longitudinal fibrous microstructure. The flax stalks, being shorter than

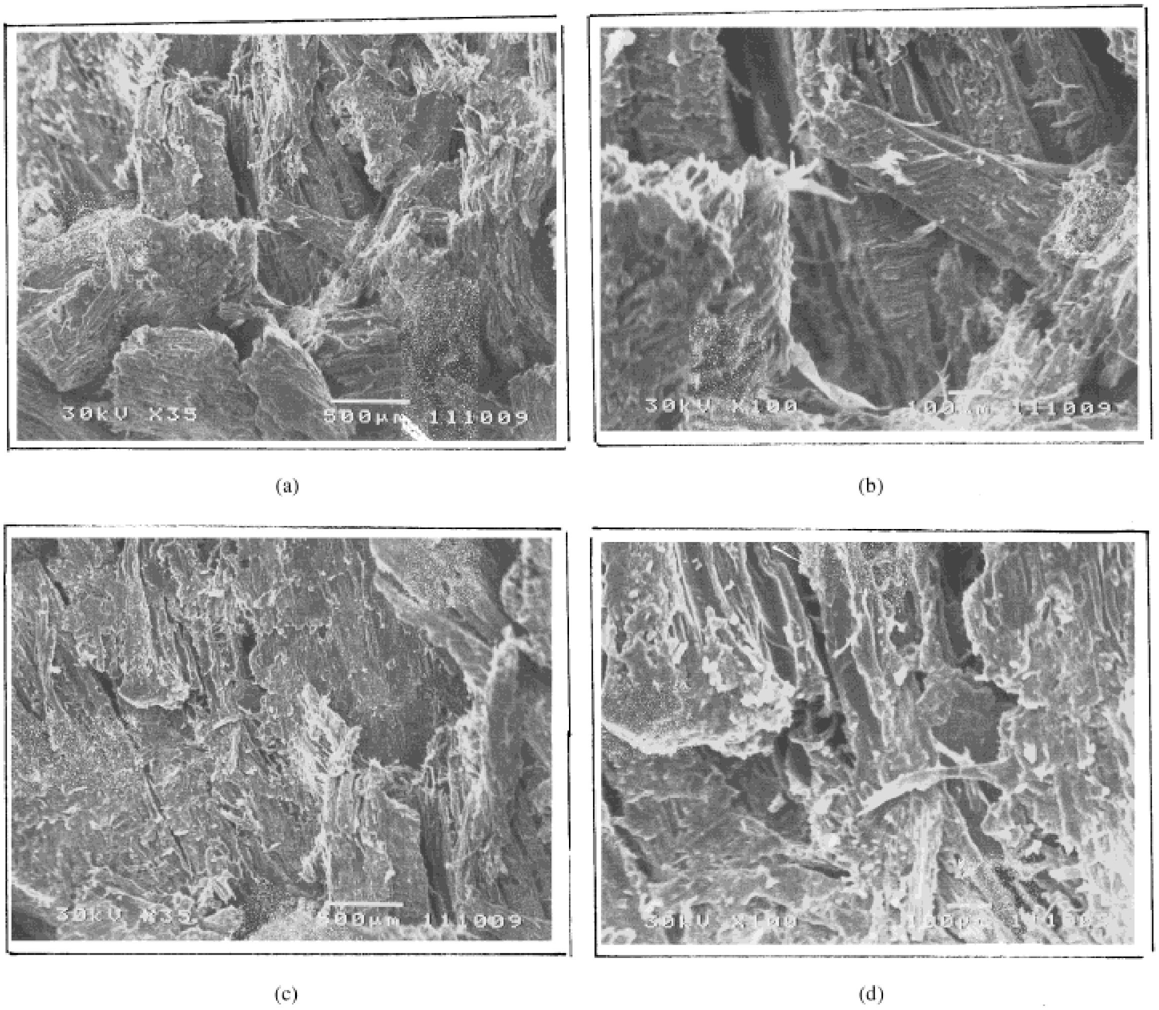


**Figure 9** SEM micrographs at different magnifications of particleboards based on wood sawdust and 20 wt % EI50: (A,B) unirradiated; (C,D) irradiated to 5 Mrad.

cotton stalks, were aligned in horizontal and perpendicular directions along the fracture surfaces. This microstructure plays a critical role in determining the mechanical properties in such a way that it makes particleboard based on cotton stalks more resistant to fracture stress. However, after gamma irradiation, the EI50 resin starts to cover and fill the distances between flax stalks as shown in Figure 7(C,D). As shown in Figure 8(A,B), the SEM micrographs of unirradiated boards indicate that the PS adhesive is relatively more compatible with the flax stalks than is the E150 resin in which the spacings and cavities are partially covered and filled. Also, the SEM micrographs of gamma-irradiated boards indicate that the spacing, pores, and cavities are covered and filled with the crosslinked PS as shown in Figure 8(C,D). However, the SEM micrographs of boards based on E150 suggest the occurrence of more grafting chains than that of PS boards.

As shown in Figure 9(A,B), the particles of wood sawdust are more compact and closer to each rather than are cotton and flax stalks even prior to gamma irradiation. After exposure to gamma irradiation, the empty areas between wood sawdust are diminished, coated, and filled with a layer of crosslinked EI50 by irradiation. Finally, the fracture surfaces become smoother due to the homogeneous distribution of the resin across the board.

Unlike cotton and flax stalks, boards based on wood sawdust and PS are more homogeneous with fewer cavities as shown in Figure 10(A,B). On the other hand, the SEM micrographs of gamma-irradiated boards indicate complete covering and filling of the spacings and cavities between

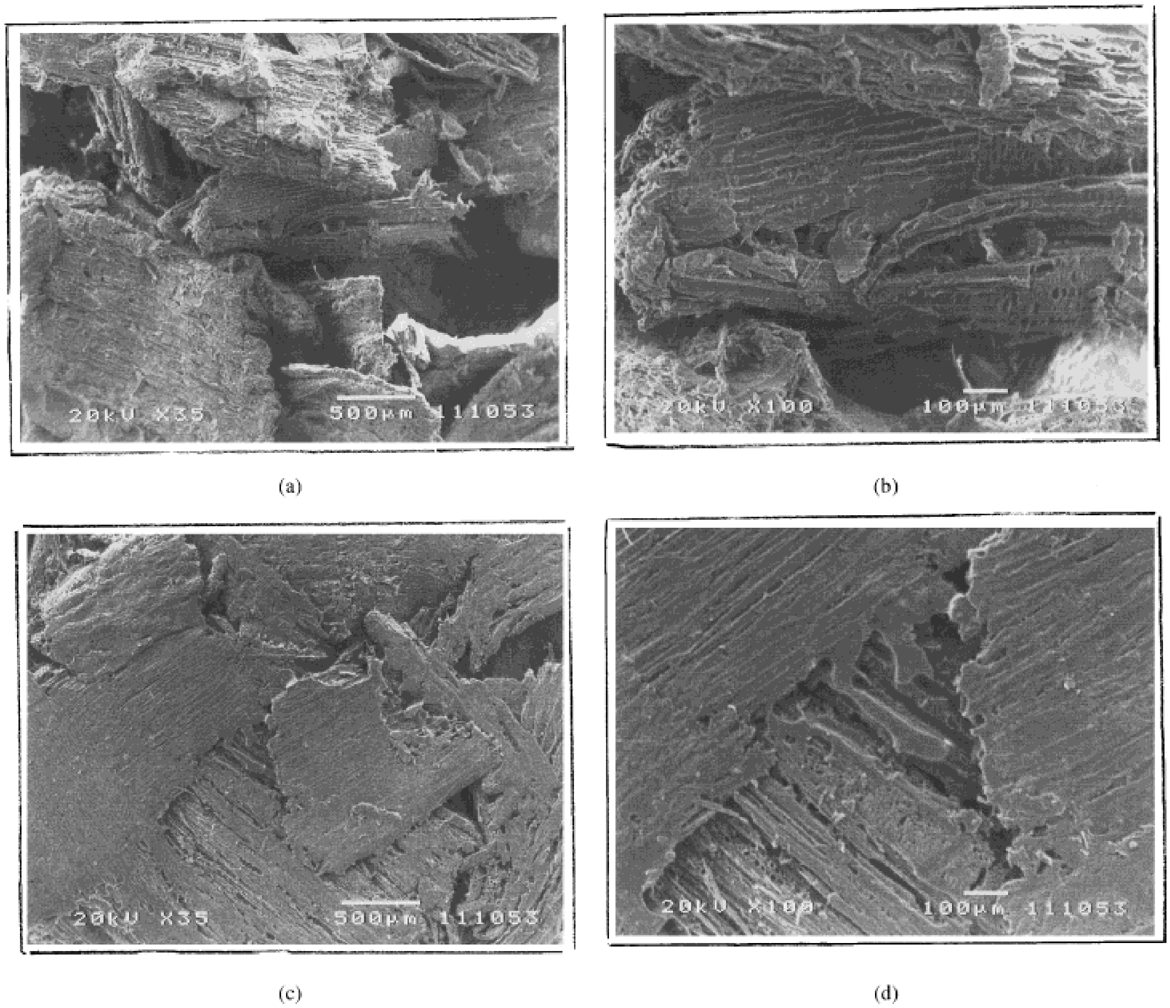


**Figure 10** SEM micrographs at different magnifications of particleboards based on wood sawdust and 20 wt % PS: (A,B) unirradiated; (C.D) irradiated to 5 Mrad.

the sawdust particles, even more than in the case of cotton and flax stalks as shown in Figure 10(C,D).

## CONCLUSIONS

The objective of this work was to study the possible fixation of polymeric adhesives inside the bulk of particleboards through crosslinking and graft copolymerization initiated by gammna irradiation. This is in addition to the ordinary thermal curing. By this method, the adhesive becomes a part of the particleboard structure, which leads to the control of the resin release. Based on the experimental results obtained from TGA and SEM observations, several points can be addressed: (1) Gamma irradiation improves, to some extent, the thermal stability of the particleboards; the improvement was found to depend on the type of waste material as well as on the type of used polymeric adhesive. This was evident from the loss in weight at different heating temperatures and the temperatures of the maximum values of the rate of reaction as determined from the derivative of the TGA curves. (2) Within the heating temperature range of 100–300°C, particleboards based on flax stalks and PS as an adhesive displayed the highest thermal decomposition over the other particleboards based on the same adhesive. However, particleboards based on wood sawdust and EI50 resin irradiated to 5 Mrad showed a significant thermal stability over the other boards based on either PS or EI50. (3) SEM observations of the fracture surfaces of the different particleboards revealed that the pores and distances between the different waste materials were covered and filled with the EI50 resin after exposure to gamma irradiation. These findings indicate that the excess resin may undergo radiation crosslinking and/or graft polymerization.

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