



CHAPTER V

RESULTS AND DISCUSSION

The effects of gamma irradiation on woodflour, polypropylene (PP), and polypropylene wood composites with and without adding maleic anhydride graft polypropylene (MA-g-PP) on the mechanical, thermal, and morphological properties were studied. The wood composite samples were prepared by a twin screw extruder at 170°C with the screw speed of 50 rpm. The filled PP wood composites were tested for the following properties; tension, flexural and creep properties. The microstructure of flexural fracture of the composites was observed by a scanning electron microscope. Thermal properties and gel contents of the samples were also investigated.

5.1 Effect of Gamma Ray on Polypropylene (PP) Matrix

5.1.1 Physical Characterization

The effects of gamma radiation on color change of polypropylene were shown in Figure 5.1. The neat polypropylene shows discoloration upon irradiation at radiation doses lower than 30 kGy. Paolo and Smith reported that polypropylene can be discolored to yellow at high irradiation. Most commercial polypropylene contains phenolic antioxidants which can turn yellow upon irradiation at 30 kGy or higher. Phenolic antioxidants produce stable phenoxyl radicals that will convert into colored quinonoids (Bentrude, 1965; De Paolo and Smith, 1968).

5.1.2 Density of Polypropylene Matrix

The measured density of all specimens was calculated using equation 4.1. The comparison of the density of polypropylene between the measured and the theoretical values were shown in Table 5.1. The measured density of the polypropylene at room

temperature was $0.9059 \pm 0.001 \text{ g/cm}^3$. The value is close to the data obtained from HMC Polymers Company LTD. The results also suggested that the density of the polypropylene only marginally increased with an increase of radiation doses. This effect demonstrated that the density rises as a result of the structural changes in the polymer, i.e. the formation of double bonds and crosslinks when irradiated with gamma ray. Similar phenomenon was also observed by Reyes et al. (Reyes, et al., 2001).

5.1.3 Mechanical Characterizations

5.1.3.1 Flexural Property Measurement

Figure 5.2 shows the effect of gamma radiation on flexural property of polypropylene which was irradiated in air and in nitrogen atmosphere. It was suggested that irradiation conditions slightly influenced the flexural properties of polypropylene. Polypropylene irradiated in nitrogen atmosphere has the flexural properties slightly higher than that irradiated in air. This irradiation caused the photons to penetrate the material, breaking the polymer chains, and creating free radicals. In addition, irradiation in air atmosphere implied the present of oxygen which may involve in the reaction. The free radical from gamma radiation can react with oxygen and generates oxidative degradation of polypropylene. This leads to a decrease of the flexural property. On the other hand, polypropylene irradiated in nitrogen atmosphere provided less oxidative degradation than that irradiated in air atmosphere since nitrogen is an inert gas that cannot react with other substances. These results were in good agreement with those reported by Al-Maadeed et al, 2006. Moreover, the effect of gamma radiation doses in nitrogen atmosphere was found to enhance the flexural modulus of the polypropylene matrix from the value of 1.18 GPa to the value of 1.45 GPa at the radiation dose of 10 kGy. Increasing the dose beyond 10 kGy, the flexural modulus was observed to be unchanged with the applied radiation dose. Similar characteristic was also observed in the flexural strength of the polypropylene irradiated with different doses of gamma ray. However, the optimal strength value was found at slightly lower radiation dose of about 5 kGy. The similar

result was reported by Reyes et al, 2001. The enhancement of flexural strength of polypropylene from the value of 46.4 MPa to the value of 55.8 MPa at radiation dose of 5 kGy was clearly observed. Using greater radiation doses has only marginal effect on the polypropylene strength i.e. 56.2 MPa at 30 kGy used. Therefore, in order to sufficiently improve the flexural properties of the polypropylene, the radiation dose of less than 10 kGy should be used. In other word, the change in the mechanical properties of the polypropylene required a relatively low level of gamma radiation which was confirmed in the studies of Yoshii et al, 1998.

5.1.3.2 Tension Property Evaluation

Polypropylene has been reported to suffer both crosslinking and chain scission at small doses of about 50 kGy, with the predominance of crosslinking (Reyes et al., 2001). Therefore, the polypropylene's molecular weight should be higher with gamma radiation resulting in an increase of the modulus and the strength as mentioned above. At greater doses, the effect of chain scission is more pronounced especially on the surface of the specimen and at the presence of oxygen. This rather high dose condition might lead to a decrease of the modulus and strength of polypropylene as observed by Reyes et al, 2001. In this work, the competition between crosslinking and chain scission reactions took place in a comparable level as the applied radiation doses were controlled to be below 30 kGy under nitrogen atmosphere. The relatively stable modulus and strength at the radiation doses of 10 to 30 kGy were observed. Figure 5.3 exhibits the tensile modulus and strength of the polypropylene at various doses of gamma radiation. The tensile results also clearly confirm that the optimal dose of the gamma radiation on the polypropylene matrix is in the ranges of 5 to 10 kGy. The tensile modulus of polypropylene irradiated in nitrogen atmosphere increased from 1.12 GPa to 1.21 GPa when irradiated at a radiation dose of 10 kGy. And then, it provided rather unchanged modulus value at higher doses. The tensile strength of the polypropylene was observed to increase to a maximum value at a dose up to 5 kGy (from 34 MPa to 34.6 MPa) of the irradiation and then it started to gradually decrease. This behavior is due to the fact that polypropylene first suffers crosslinks at low irradiation dose, which confers a greater

resistance at break, but at higher irradiation doses, a slight decrease in the resistance to break was observed.

In addition, the Figure 5.3 exhibits the tensile properties of polypropylene that irradiated in air and nitrogen atmospheres. It indicated that the tensile properties of irradiated polypropylene in air are close to the irradiated polypropylene in nitrogen atmosphere.

5.1.3.3 Creep Property Measurement

Figure 5.4 shows the creep behavior of irradiated polypropylene at various doses as a function of time. The stresses used corresponded to the value 40% of the tensile strength of neat polypropylene. All specimens showed permanent irrecoverable strain even though the applied load was well within the linear viscoelastic region. This behavior suggested the non-linear behavior of all specimens. As shown in Figure 5.4, the creep up to six hour duration under tension of the neat polypropylene indicated the values slightly decreased with increasing the radiation doses. The gamma irradiation can improve the creep behavior for approximately 14% in 30 kGy irradiation of polypropylene. The fracture surface of these radiation-treated samples also revealed a smoother surface as signature of a more rigid nature of the irradiated samples.

5.1.4 Thermal Characterizations

5.1.4.1 Differential Scanning Calorimetry (DSC)

Melting temperature of the polymer matrix can be determined by Differential Scanning Calorimetry. Figure 5.5 presents DSC thermograms of the second heating of polypropylene subjected to different irradiation doses. Only one endothermic peak corresponding to polypropylene melting can be observed in this figure. The temperature at the DSC endothermic peak gives the value of melting temperature (T_m) of polypropylene. Those values are listed in Table 5.2. We can see that, the T_m values were relatively constant at a radiation dose in the range of 0 to 30

kGy. Similar characteristic was also observed by Albano et al., 2001 who demonstrated that gamma ray treatment at low radiation dose between 0 and 25 kGy provided relatively constant value of the melting temperature. T_m was observed to decrease at higher doses. This behavior was possibly caused by the oxidative degradation of polypropylene when it was exposed to high irradiation dose, which promoted chain scission of polymer molecules.

5.1.4.2 Thermogravimetric Analysis (TGA)

Figure 5.6 shows the decomposition behavior of polypropylene obtained from TGA experiment. All the irradiated polypropylene, at different irradiation doses, possessed only one decomposition stage as seen in the thermograms. The gamma irradiation significantly affected on the degradation temperature at 10% weight loss of polypropylene as summarized in Table 5.3. The degradation temperature decreased with increasing the radiation dose as the degradation temperature of the non-irradiated polypropylene is approximately 448°C. Another important feature in the thermograms is the percent residue at 800°C of the irradiated polypropylene. Figure 5.6 and Table 5.3 show the char yield, one of the parameters related to the material flame resistance, of the irradiated polypropylene at radiation doses ranging from 0 to 30 kGy indicating a relatively constant value with increasing the radiation. However, the value was slightly lower than that of untreated polypropylene. For example, the char yield of a non-irradiated polypropylene was 1.43% and the value decreased to 0.24% in the irradiated polypropylene at about 5 kGy of radiation dose.

5.1.5 Interfacial Interaction

Adhesion at the interface of organic fillers and polymers is one of the main factors determining the properties of polymer composites. Under an influence of modifier molecules, the resulting improvement of the physical-mechanical properties of wood composites such as strength and young's modulus has been reported (Qiu, 2005; Sompatsompop, et al., 2005). The interfacial interaction can be qualitatively

examined using a scanning electron micrograph. Figure 5.7 represents SEM micrographs of fracture surfaces of polypropylene specimens irradiated with gamma ray in nitrogen at 0 kGy, 10 kGy, and 30 kGy. We can see that, the gamma radiation has significant effect on morphological behavior of the polypropylene. The non-irradiated polypropylene had a lot of yielding behavior and behavior roughness on the fracture surface while irradiated polypropylene at 10 and 30 kGy showed a smoother and a more glass-like fracture surface. In addition, the irradiated polypropylene at 30 kGy indicated the smoothest fracture surface which suggested the change from a more ductile to a more brittle material upon gamma radiation treatment probably due to the crosslinking reaction.

5.2 Effect of Gamma Ray on *Hevea Brasiliensis* Woodflour

5.2.1 Physical Characterization

The filler used in this investigation is rubber woodflour (*Hevea brasiliensis*), having an average particle size of 250-300 micrometer with an aspect ratio of about 3 as shown in Figure 5.8. The gamma irradiation had some effects on the physical characterizations of rubber wood such as causing acrid odour when treated at high radiation dose up to 30 kGy. This phenomenon could demonstrate that gamma radiation might cause the degradation of the rubber wood.

5.2.2 Mechanical Characterizations

5.2.2.1 Flexural and Tension Property Measurement

In order to understand the effect of gamma radiation on polypropylene wood composites more clearly, effects of gamma radiation on flexural properties of *Hevea Braziliensis* wood, which was used as filler, were also examined and the results were depicted in Figure 5.9-5.10. From these figure, the flexural modulus and strength of the wood was found to initially increase with increasing the radiation doses in the range of 0-10 kGy. After reaching the maximum values at the dose of 10 kGy, both

modulus and strength of the wood decrease steadily with increasing the radiation doses. The flexural modulus of the rubber wood was determined to be 7.8 GPa and was raised to 9.6 GPa when it was irradiated at the 10 kGy level. In addition, from Figure 5.10, the flexural strength of the wood material was 117 MPa and was increased to 148 MPa after the irradiated with 10 kGy of gamma radiation. The phenomenon, therefore, suggests the crosslinking reaction in the wood material to overcome the chain scission reaction when the radiation dose was maintained to be less than 10 kGy. As a consequence, the radiation level of less than 10 kGy should be applied to the polypropylene wood composites in order to maintain their mechanical integrity with negligible degradation of the properties of the polypropylene matrix and the woodflour filler.

5.2.3 Thermal Properties

5.2.3.1 Thermogravimetric Analysis (TGA)

TGA Thermogram shows the thermal property of rubber wood (*Hevea brasiliensis*) as shown in Figure 5.11. From this figure, it could be concluded that the rubber woodflour has the degradation temperature about 260°C at 10% weight loss. The thermal degradation of rubber wood was a two-stage process, one in the temperature range of 220-280°C and another in the range 360-430°C. The low temperature degradation process was associated with degradation of hemicellulose and the cleavage of the glycosidic linkages of cellulose whereas the high temperature process was due to lignin (Saheb and Jog, 1999; Jubsilp, et al., 2006). In addition, the temperature about 100°C on TGA thermogram was assigned to the vaporization of water in the woodflour. All the hydroxyl and carboxylic groups in the woodflour were capable of absorbing moisture. The degradation behavior of natural wood had been studied by Jubsilp et al. and Saheb et al.

5.3 Effects of Gamma Ray on Polypropylene *Hevea brasiliensis* Woodflour Composites

5.3.1 Physical Characterization

From Figure 5.12, the polypropylene woodflour composites exhibit dark brown color with some rough surface texture of woodflour. In addition, it gave off acrid odour when irradiated with gamma radiation at high radiation dose approximate 30 kGy. The odour was probably caused by the decomposition of some components in the rubber wood.

5.3.2 Density of Polypropylene Woodflour Composites

The measured density of polypropylene woodflour composites was calculated using equation 4.1 and the theoretical density was tabulated in Appendix A. Table 5.4 shows the comparison of theoretical density and measured density of the polypropylene wood. The densities of all wood composite specimens are about 1.064 g/cm³ which are higher than that of the neat polypropylene i.e. 0.906 g/cm³. Whereas the woodflour used has the measured of density 1.49 g/cm³. In addition, this table suggests that gamma irradiation had negligible effect on the density of the wood composites and their theoretical densities are close to the measured densities. Therefore, the fabrication of these composites introduced no void or at least minimal void content in the composite.

5.3.3 Mechanical Characterizations

5.3.3.1 Flexural Property Measurement

The general problem in using wood fiber together with a thermoplastic polymer is the occurrence of agglomeration due to insufficient dispersion in high viscous polymer matrix. Therefore, wood or cellulose fiber can be incorporated into a

thermoplastic polymer of less than 50-60% by weight normally with certain degree of difficulty. Figure 5.13 illustrates the effect of the gamma radiation on flexural properties of polypropylene woodflour composites, which irradiated under both air and nitrogen atmospheres. The polypropylene woodflour composites exhibited an increase in the flexural properties as function of gamma radiation up to the radiation dose of 10 kGy. Then, the properties started to decrease. Under nitrogen atmosphere, the flexural modulus of irradiated composite at radiation dose of 10 kGy was found to be 3.5 GPa, which is higher than that of non-irradiated polypropylene woodflour composites having the flexural modulus of 2.9 GPa. Then, the value decreased beyond the irradiation dose of 10 KGy. This behavior may be due to the fact that the polypropylene and woodflour had generated a predominant crosslinking at low radiation dose. In addition, gamma radiation can lead to interaction between polypropylene matrix and woodflour to a degree greater than non-irradiated composites. This behavior was confirmed by SEM micrographs of the polypropylene woodflour composites. In addition, the interfacial of interaction of the irradiated composites was explained in the studies of Albano et al., 2001-2002. Similar characteristic was also observed in the flexural strength of the polypropylene woodflour composites irradiated with different doses of gamma ray. The flexural strength of the composites was found to increase from the value of 48.6 MPa to the value of 50.9 MPa at the radiation dose of 10 kGy.

In addition, Figure 5.13 depicts the effect of gamma radiation on polypropylene woodflour composites in presence of both air and nitrogen atmospheres. From the previous work, irradiation in air causes the formation of free radicals, which react with oxygen. This phenomenon generates oxidative degradation leading to scission of the main chain. Al-Maadeed et al., 2006 reported that oxidative degradation due to gamma irradiation in an inert gas such as nitrogen at low irradiation dose (lower than 200 kGy) was negligible.

5.3.3.2 Tensile Property Evaluation

Tensile properties of our polypropylene wood composites irradiated with gamma ray are also illustrated in Figure 5.14. The polypropylene wood irradiated

in nitrogen atmosphere revealed an enhancement in its tensile modulus and tensile strength when the radiation doses were kept below 10 to 20 kGy confirming our studies on the influences of the matrix and the filler with the gamma radiation in the previous section. The radiation doses above 20 kGy evidently showed a negative effect on both properties by substantially lowering those values. The phenomenon is attributed to the strong effect of gamma radiation to deteriorate the mechanical properties of the wood filler comparing with the effect on those of neat polypropylene matrix as seen in Figure 5.14. The tensile modulus of the polypropylene wood (at 40% by weight of woodflour) was measured to be 2.29 GPa and increased to 2.53 GPa after irradiation with 10 kGy of the gamma radiation whereas the tensile strength increased from 28.6 MPa of the non-irradiated specimen to the value of 29.5 MPa using 10 kGy of the radiation.

The increase in the mechanical properties of the polypropylene wood at relatively low range of gamma radiation dose may be attributed to the slight crosslinking of the polypropylene as well as of the woodflour filler. Furthermore, Albano et al., 2001-2002 reported the higher polymer-filler interaction due to an increase in hydrophilicity of the polypropylene after irradiation by gamma ray. The authors also observed the sign of H-bonding between the woodflour and the irradiated polypropylene in infrared spectra. A significant decrease in both modulus and strength of the polypropylene wood composites beyond 10 kGy of the applied gamma radiation should mainly be caused by the degradation of the woodflour filler. In practice, a measure to protect woodflour photodegradation from irradiation might be essential in order to maintain a polypropylene wood composite's mechanical integrity. Moreover, the effect of the change of irradiation atmosphere from nitrogen to air shows negligible influence on the values of both tensile modulus and strength in our polypropylene wood composite.

5.3.3.3 Creep Property Measurement

The comparison of creep behaviors under tensile loading between the polypropylene and woodflour filled polypropylene composites was exhibited in Figure 5.15. It is apparent that the effect of woodflour content on creep is relatively

significant. The tensile creep strain decreased with an addition of the woodflour. The creep strain of the composite filled with woodflour at 40% by weight and at the 40% stress level was significantly lower than that of the creep strain of the neat polypropylene. It was found that the addition of 40% by weight of woodflour as a filler in polypropylene composite could improve the creep behavior of approximately 54%.

The effects of gamma radiation at various irradiation doses on the creep behaviors of the polypropylene wood composites were shown in Figure 5.16. The figure indicated that gamma irradiation had some effect on creep properties of woodflour composites. The tensile creep strain of the woodflour composite was found to initially decrease with increasing the radiation doses within the range of 0 to 10 kGy. After that the creep values decreased as a function of radiation doses. We could conclude that the gamma irradiation at low radiation below 10 kGy could improve the creep resistance of the woodflour composites because it enhanced the compatibility between the woodflour filler and the polypropylene matrix. However, the woodflour tended to degrade at higher radiation doses i.e. above 20 to 30 kGy that led to poor creep resistant properties.

5.3.4 Thermal Characterizations

5.3.4.1 Differential Scanning Calorimetry (DSC)

Table 5.5 and Figure 5.17 exhibit DSC characteristics of polypropylene filled with 40% by weight of woodflour at various radiation dose rates. From this figure, all specimens of woodflour composites showed only one endothermic peak of the melting phenomenon. Polypropylene woodflour composite possess a melting temperature of 165°C while the melting temperatures of all irradiated polypropylene woodflour composites slightly decreased with the radiation doses. This behavior may be caused by the chain scission reaction due to oxidative degradation of the polypropylene when it was subjected to gamma radiation.

5.3.4.2 Thermogravimetric Analysis (TGA)

Figure 5.18 shows decomposition behaviors of the irradiated polypropylene woodflour composites at different irradiation doses. Three decomposition stages can be identified, those in the temperature range of 260-320°C and 360-430°C were reported to be the degradation of hemicellulose and lignin, respectively, another one in the range of 430-460°C was associated with the degradation of the polypropylene. The polypropylene/woodflour composite had the decomposition thermogram with the combined characteristics of the woodflour and polypropylene which was typical for heterogeneous materials. The corresponding degradation temperature at 10% weight loss and char yield values were also listed in Table 5.6. This table revealed that gamma irradiation had negligible effect on the degradation temperature and char yield of the polypropylene. Though crosslinking might be occurred at low radiation doses as discussed previously, that low degree of crosslinking showed negligible effect studied on the degradation temperature and char yield of our polypropylene wood.

5.3.5 Interfacial Interaction

SEM micrographs of the fracture surface of the polypropylene woodflour composites at radiation doses of 0, 10, and 30 kGy are shown in Figure 5.19. In general, we know that the woodflour was not compatible with polypropylene because of the nature of woodflour is hydrophilic while polypropylene is hydrophobic. From Figure 5.19, we could see that the gamma irradiation had some effect on the polymer-filler interaction. The micrograph of the non-irradiated wood composites showed rather poor adhesion that had holes and fissures between the filler and the matrix. Whereas the micrographs of the irradiated wood composites showed substantial adhesion between the two phases. Though, irradiation at higher radiation dose seems to generate greater interfacial adhesion between the polypropylene matrix and the woodflour as seen from Figure 5.19, at this high dose level, the woodflour tended to degrade that led to the decrease of the mechanical properties of the woodflour composite as discussed in Figure 5.12 and 5.13.

5.4 Effects of Gamma Ray on Polypropylene/*Hevea brasiliensis* Woodflour Composites with and without a Compatibilizer

5.4.1 Physical Characterization

In this part, a compatibilizer, maleic anhydride graft polypropylene (PP-g-MA), at 3% by weight of woodflour was selected for further investigation. The concentration was reported as an optimal value to achieve good wood composite properties (Sombatsompop, et al., 2005; Lee, et al., 2004). Figure 5.20 shows physical characterization of our woodflour composites mixed with PP-g-MA and irradiated with gamma ray at various doses. Compared with Figure 5.12, the polypropylene woodflour composites treated with a PP-g-MA, at 3% by weight of woodflour, had the physical characterizations similar to the untreated woodflour composites. In addition, the wood composite having PP-g-MA and being treated with gamma radiation had some observable changes in the properties. It gave acrid odour possibly caused by the degradation of some components in the woodflour or PP-g-MA when irradiated with gamma irradiation.

5.4.2 Density of Polypropylene Woodflour Composites with PP-g-MA

The void content in polypropylene woodflour composites treated with PP-g-MA followed by gamma radiation was evaluated by the density measurement. The density of woodflour composites treated with a compatibilizer was calculated using equation 4.1 and compared with the theoretical density was tabulated in Appendix A. Table 5.7 shows the comparison between theoretical and measured densities of woodflour composite treated with PP-g-MA and irradiated with gamma ray at various radiation doses. From the table, the density of woodflour composites treated with PP-g-MA was determined to be 1.062 g/cm^3 which are slightly lower than that of woodflour composites without PP-g-MA i.e. 1.064 g/cm^3 . This is because the density of PP-g-MA was 0.94 g/cm^3 . Therefore, the density of the composite with PP-g-MA should be lower than the composite without PP-g-MA. In addition, Table 5.7 reveals

that the measured densities of irradiated composites at various doses are relatively close to their theoretical densities. This behavior indicated that these composites at least possessed minimal void content in their system. Furthermore, this table suggests that the gamma radiation at various doses had no significant effect on the densities of the composites.

5.4.3 Mechanical Characterizations

5.4.3.1 Flexural and Tensile Property Measurement

As it was pointed out in Section 5.3, the effects of gamma radiation on woodflour composites provided some enhancement in the mechanical and morphological properties at a suitable range of radiation doses. For example, the flexural modulus of polypropylene woodflour composites irradiated with gamma ray at the radiation dose of 10 kGy displayed approximately 22.3% increase when compared with the unirradiated samples. In Section 5.4, the compatibilizer, maleic anhydride graft polypropylene (PP-g-MA), was introduced into the irradiated woodflour composites for improving the interfacial adhesion between the woodflour filler and the polypropylene matrix. Figure 5.21 exhibits the effect of gamma radiation at various radiation doses in combination with the treatment with PP-g-MA at 3% by weight of woodflour on flexural properties of woodflour composites. From Figures 5.21 and 5.13, the effect of PP-g-MA was found to significantly improve the flexural strength of woodflour composites from the value of 48.6 MPa to the value of 57.1 MPa whereas it was no significantly effect on the flexural modulus. In addition, Figure 5.21 shows that gamma irradiation on the woodflour composites treated with PP-g-MA had some effect on the flexural modulus by increasing its value from 2.87 GPa to 3.51 GPa at the radiation of 10 kGy. The radiation, however, was found to show negligible effect on the composites flexural strength.

As aforementioned, we conclude that the PP-g-MA had negligible effect on the flexural modulus of the composites but it had an observable effect on the flexural strength of these composites. The flexural modulus of the composites increased when the gamma irradiation at optimal radiation doses was applied in the

composite with PP-g-MA treatment. This effect suggested that gamma radiation might also cause some changes in the molecules of PP-g-MA. Because PP-g-MA has a carbonyl group that is unstable with gamma irradiation. This behavior might cause the scission of the molecules of PP-g-MA that led to decreasing of the flexural strength. Similar characteristic was also observed in the tensile modulus and tensile strength of these PP-g-MA added composites and irradiated with different doses of gamma ray as depicted in Figure 5.22.

5.4.3.2 Creep Property Measurement

Figure 5.23 shows the effect of gamma irradiation at various doses on creep behavior of polypropylene/wood composites with and without PP-g-MA. The effect of PP-g-MA on creep was found to be relatively significant. The addition 3% by weight of PP-g-MA as a compatibilizer in polypropylene woodflour composite could improve the creep behavior of approximately 20%. Moreover, this figure shows that the combined effect of PP-g-MA and gamma irradiation on wood composites could further improve creep resistance when the radiation was less than 10 kGy while greater irradiation doses suggested greater the tensile creep strain. We could conclude that the effect of PP-g-MA combined with gamma radiation led to the decrease of creep behavior for approximately 36%.

5.4.4 Extraction Percentages Examination

The gel content of polypropylene and its composites with and without the addition of PP-g-MA was determined using xylene extraction according to ASTM D 2765 (method C). Crosslinked polypropylene and its composites at different radiation doses were insoluble in hot xylene while the un-crosslinked part remained soluble. The extraction percentages were calculated using Equation 4.3 and these values were tabulated in Appendix C. This appendix also exhibits the extraction percentage of the neat polypropylene as a function of radiation doses in the range of 0 to 10 kGy. The value of extracted content of the non-irradiated polypropylene was determined to be which was 65.7% higher than that of the irradiated polypropylene at 10 kGy having

the value of only 5.98%. Using greater radiation doses, the extraction percentage of the polypropylene was observed to be unchanged with the applied the radiation dose. This behavior revealed that the polypropylene was underwent crosslinks when it received gamma radiation, which conferred a greater resistance solubility. Moreover, the same behavior was observed in cases of its composites treated and untreated with PP-g-MA. Figure 5.24 and 5.25 show the physical characterization of irradiated polypropylene and its composite with and without PP-g-MA at different doses. They confirm the crooslinking in the polymer chain of polypropylene and increasing interfacial adhesion between woodflour filler and polypropylene matrix. Because the non-irradiated specimens were dissolved in hot xylene while irradiated specimens were partially dissolved. This phenomenon led to enhance of mechanical properties of polypropylene.

5.5 Effects of Radiation Dose Rates of Gamma Ray on Polypropylene *Hevea brasiliensis* Woodflour Composites

5.5.1 Mechanical Characterizations

5.5.1.1 Flexural and Tensile Property Measurement

In the previous part in topic 5.3 and 5.4 of this research show the effect of gamma radiation at various doses on the mechanical, thermal behavior of the polypropylene/woodflour composites with and without PP-g-MA. It could conclude that the radiation suitable for improve the properties of the composite was about 10 kGy at radiation dose rate 0.2112 kGy/min. In this part, we will find the effect of gamma radiation dose rates that suitable to improve the properties of the composites. The gamma radiation dose was fixed at 10 kGy and the radiation dose rate was related with the irradiation time. The effect of radiation dose rate that related to the mechanical properties of woodflour composites was shown in Table 5.8. The mechanical properties of the composites increased as function of radiation dose rates. The modulus and strength of the composite that irradiated with gamma ray at

radiation dose rate of 0.2112 kGy/min was highest. The enhancement of flexural modulus and flexural strength of the composite from the value of 2.54 GPa to 3.29 GPa and 39.49 MPa to 48.48 MPa, respectively, at radiation dose rate of 0.2112 kGy/min was clearly observed. In addition, the tensile modulus and tensile strength increased from the value of 1.94 GPa to 2.48 GPa and 23.24 MPa to 29.18 MPa, respectively. This phenomenon was possibly caused by the oxidative degradation, since oxygen diffuses more easily into the polypropylene for longer radiation time.

5.5.1.2 Creep Property Measurement

Figure 5.26 presents the creep curves of polypropylene woodflour composite that irradiated with gamma ray at different dose rates. This creep curves were obtained from 6 hr constant loading with 40% tensile strength of neat polypropylene. It is apparent that a significant difference in creep behavior was observed. The tensile creep strain decreased with increasing the radiation dose rate. The radiation dose rate at 0.2112 kGy/min could improve the creep behavior for approximately 13% when compared with the irradiated composite at radiation dose rate 0.0614 kGy/min. This effect was demonstrated that using lower irradiation dose rate on the composite related with using the longer radiation time that generated the greater oxidative degradation which led to the decreased of the creep resistant.

Table 5.1: The density of polypropylene irradiated with gamma rays at various doses.

Radiation doses (kGy)	Theoretical density (g/cm³)	Measured density (g/cm³)
0	0.9000	0.9059±0.001
5	0.9000	0.9061±0.001
10	0.9000	0.9061±0.001
20	0.9000	0.9068±0.001
30	0.9000	0.9077±0.001

Table 5.2: The melting temperature of polypropylene irradiated with gamma rays at various doses.

Radiation doses (kGy)	Melting temperature (°C)
0	164
5	163
10	163
20	163
30	162

Table 5.3: The degradation temperature and char yield of polypropylene irradiated with gamma rays at various doses.

Radiation doses (kGy)	Degradation temperature (°C)	Char yield (%)
0	448	1.43
5	445	0.24
10	443	0.36
20	442	0.38
30	441	0.40

Table 5.4: The density of woodflour composites irradiated with gamma rays at various doses.

Radiation doses (kGy)	Theoretical density (g/cm³)	Measured density (g/cm³)
0	1.069	1.064±0.001
5	1.069	1.064±0.001
10	1.069	1.064±0.001
20	1.069	1.063±0.001
30	1.069	1.064±0.001

Table 5.5: The melting temperature of polypropylene woodflour composites irradiated with gamma rays at various doses.

Radiation doses (kGy)	Melting temperature (°C)
0	165
5	165
10	163
20	163
30	162

Table 5.6: The degradation temperature and char yield of polypropylene woodflour composites irradiated with gamma rays at various doses.

Radiation doses (kGy)	Degradation temperature (°C)	Char yield (%)
0	322	7.48
5	322	7.73
10	322	7.50
20	322	7.73
30	322	6.26

Table 5.7: The density of PP/wood/PP-g-MA composites irradiated with gamma rays at various doses.

Radiation doses (kGy)	Theoretical density (g/cm³)	Measurement density (g/cm³)
0	1.071	1.062±0.001
5	1.071	1.062±0.001
10	1.071	1.063±0.001
20	1.071	1.063±0.000
30	1.071	1.064±0.001

Table 5.8: The mechanical properties of PP wood composites irradiated with gamma rays at various dose rates.

Dose rates (kGy/min)	Flexural modulus (GPa)	Flexural strength (MPa)	Tensile modulus (GPa)	Tensile strength (MPa)
0.0614	2.54±0.05	39.49±0.17	1.94±0.08	23.24±1.80
0.0850	2.61±0.03	39.73±0.28	2.17±0.03	24.15±0.45
0.1515	2.67±0.07	40.18±0.31	2.11±0.12	25.03±1.29
0.2112	3.29±0.04	48.48±0.57	2.48±0.05	29.18±0.67

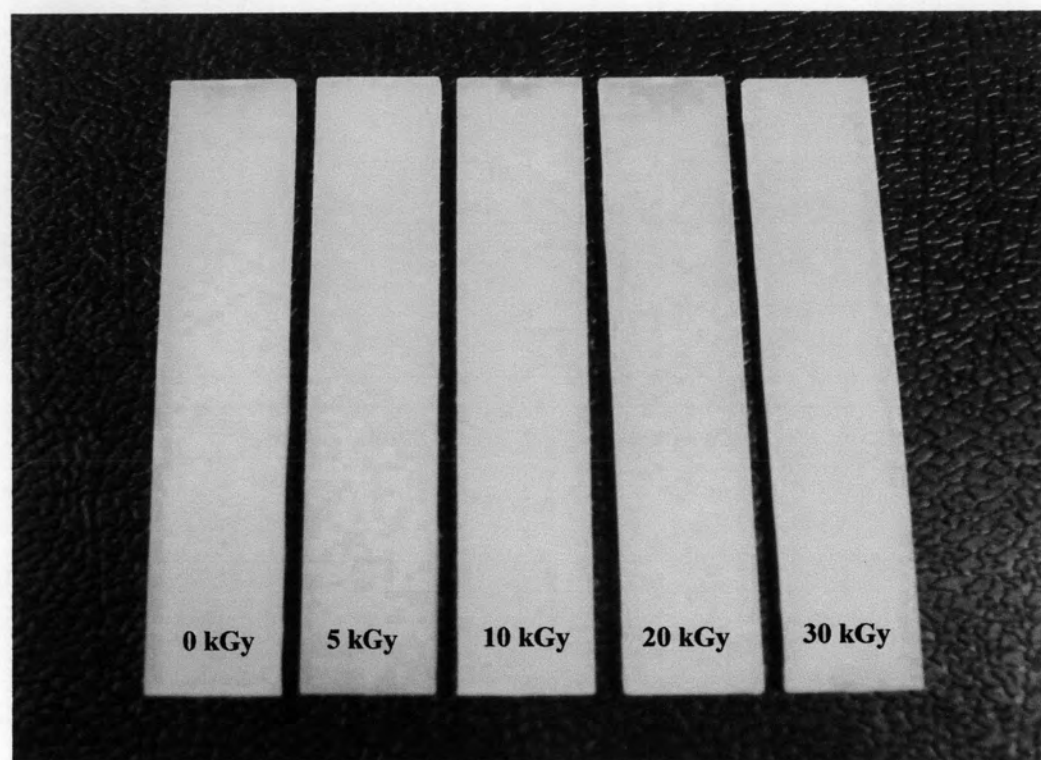


Figure 5.1: Effect of gamma irradiation on discoloration of PP.



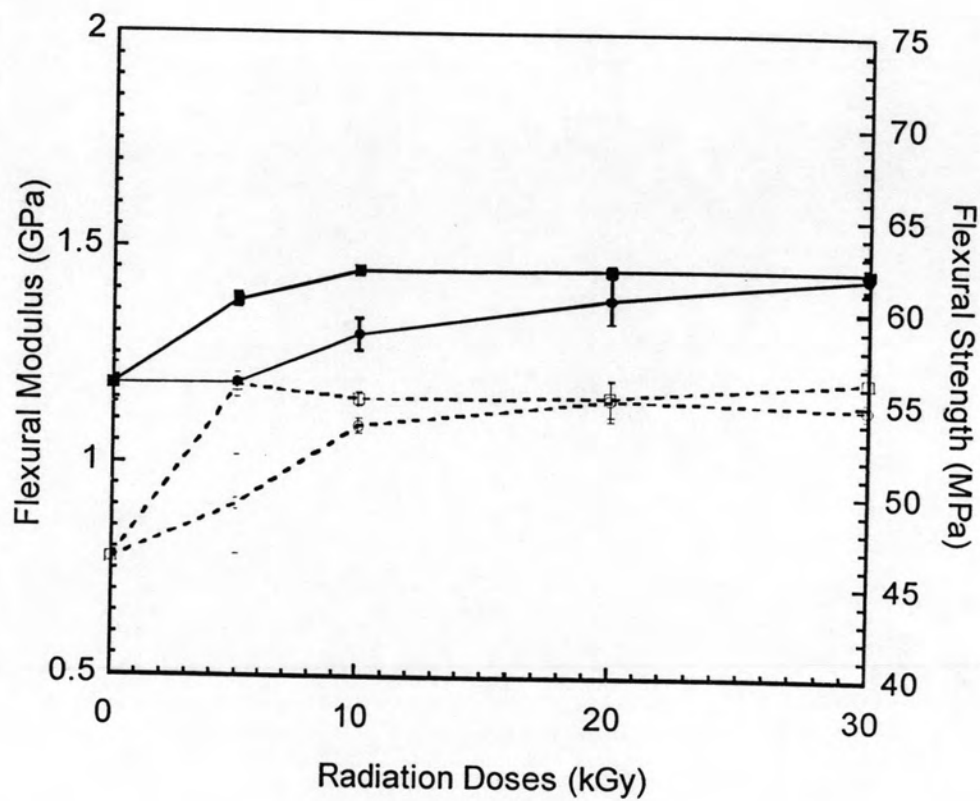


Figure 5.2: The flexural properties of polypropylene at various doses: (●) flexural modulus in air, (■) flexural modulus in nitrogen, (○) flexural strength in air, (□) flexural strength in nitrogen.

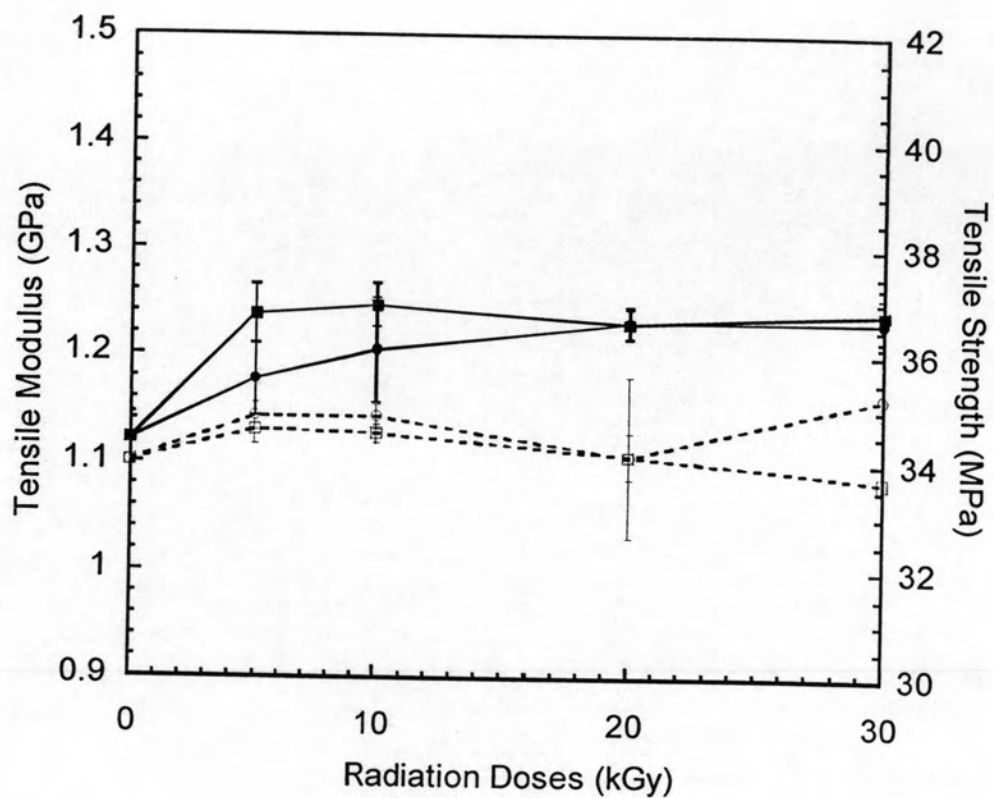


Figure 5.3: The tension properties of polypropylene at various doses: (●) tensile modulus in air, (■) tensile modulus in nitrogen, (○) tensile strength in air, (□) tensile strength in nitrogen.

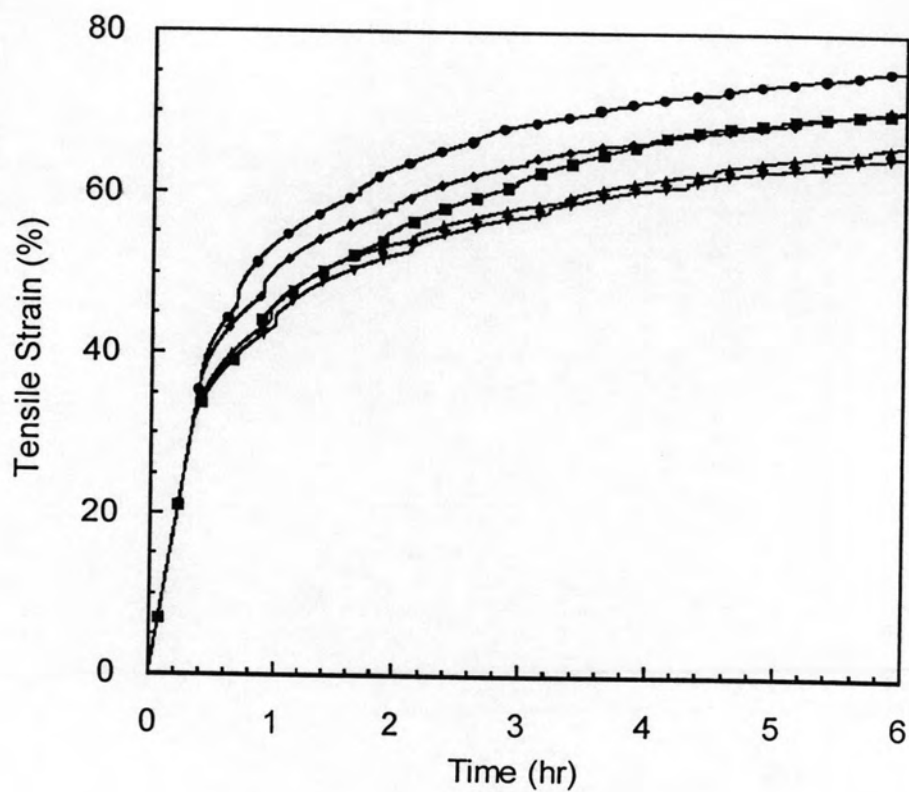


Figure 5.4: Creep behavior of polypropylene at various doses: (●) 0 kGy, (■) 5 kGy, (◆) 10 kGy, (▲) 20 kGy, (▼) 30 kGy.

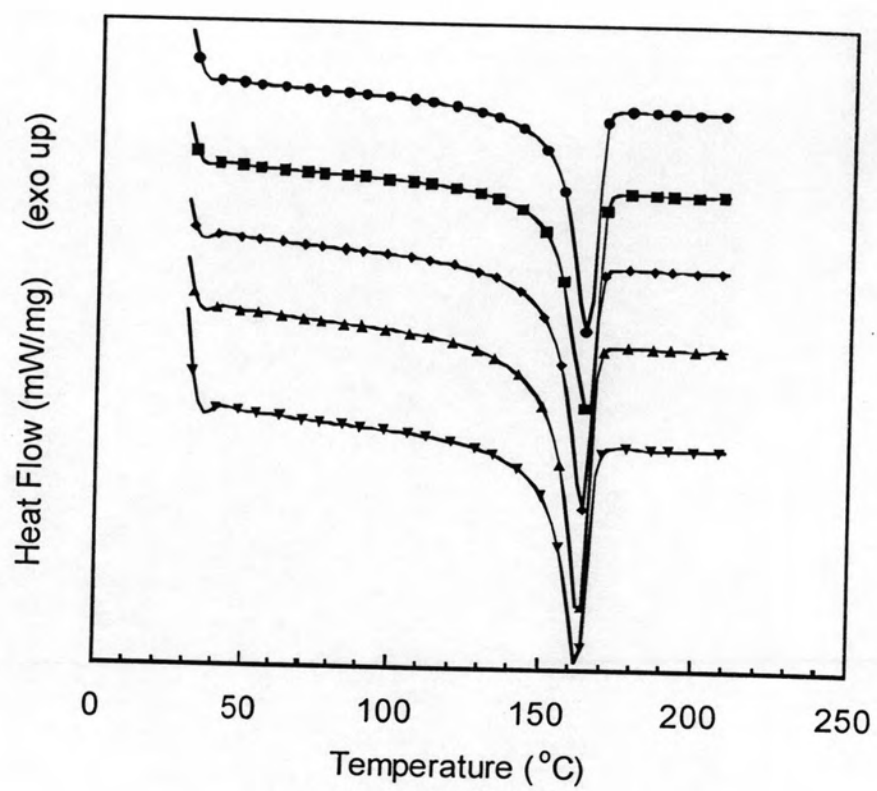


Figure 5.5: DSC thermograms of polypropylene at various doses:

(●) 0 kGy, (■) 5 kGy, (◆) 10 kGy, (▲) 20 kGy, (▼) 30 kGy.

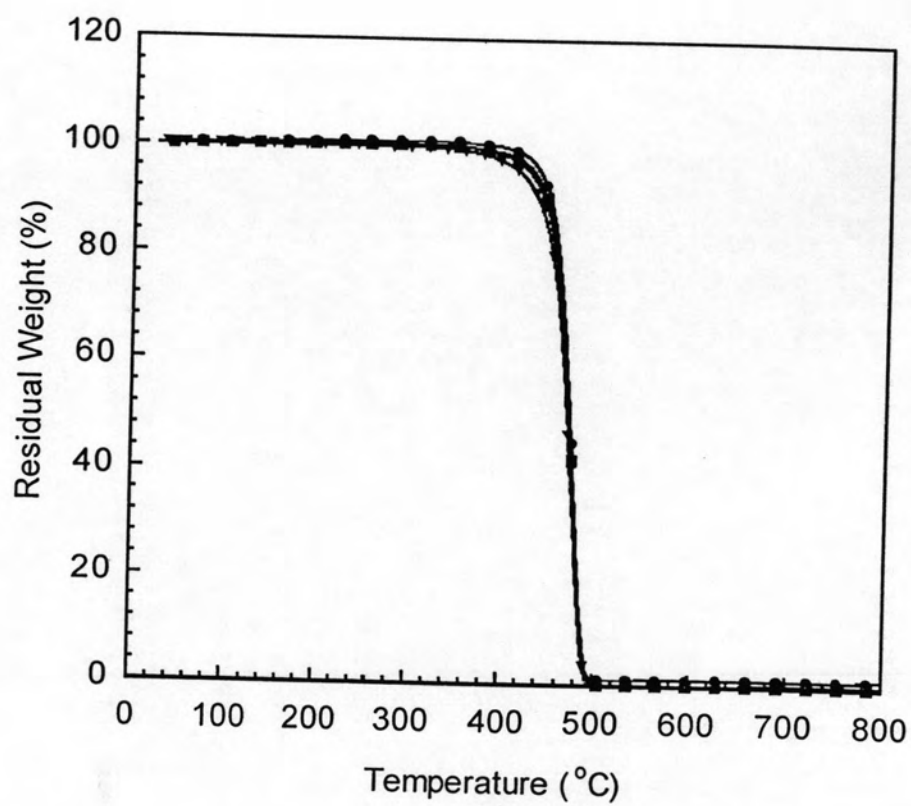
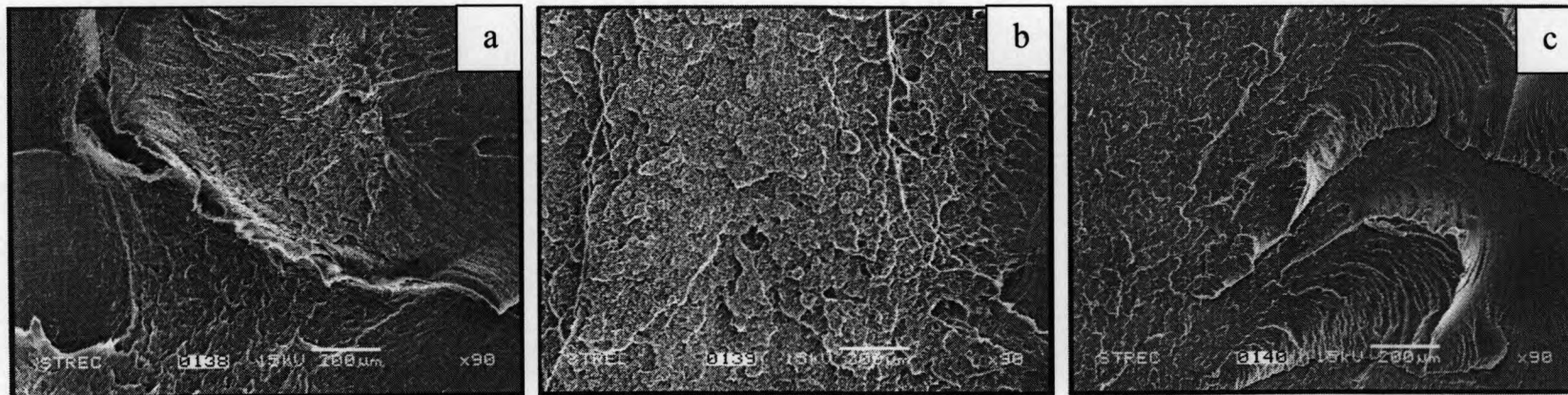


Figure 5.6: TGA thermograms of polypropylene at various doses:
(●) 0 kGy, (■) 5 kGy, (◆) 10 kGy, (▲) 20 kGy, (▼) 30 kGy.



Polypropylene

Polypropylene 10 kGy

Polypropylene 30 kGy

Figure 5.7: SEM micrographs on fracture surface of polypropylene at various radiation doses: (a) 0 kGy, (b) 10 kGy, (C) 30 kGy.

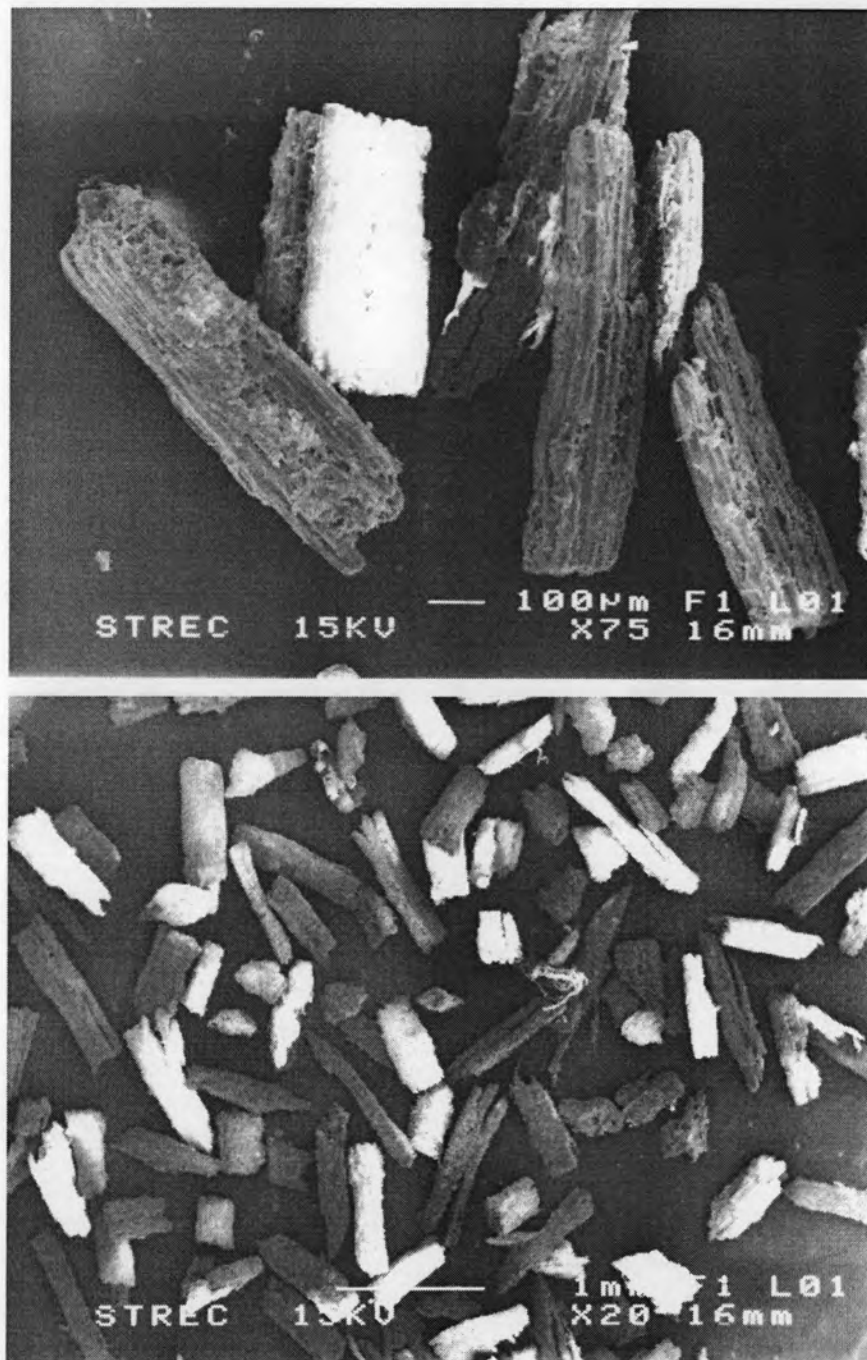


Figure 5.8: The average particles size of rubber woodflour.

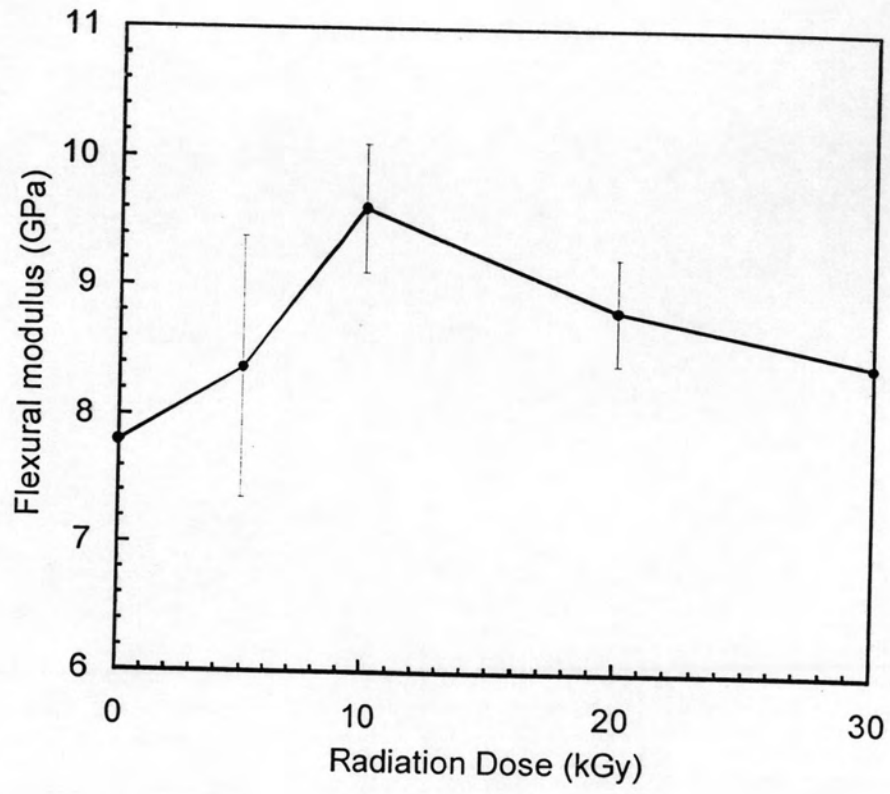


Figure 5.9: The flexural modulus of rubber wood at various doses.

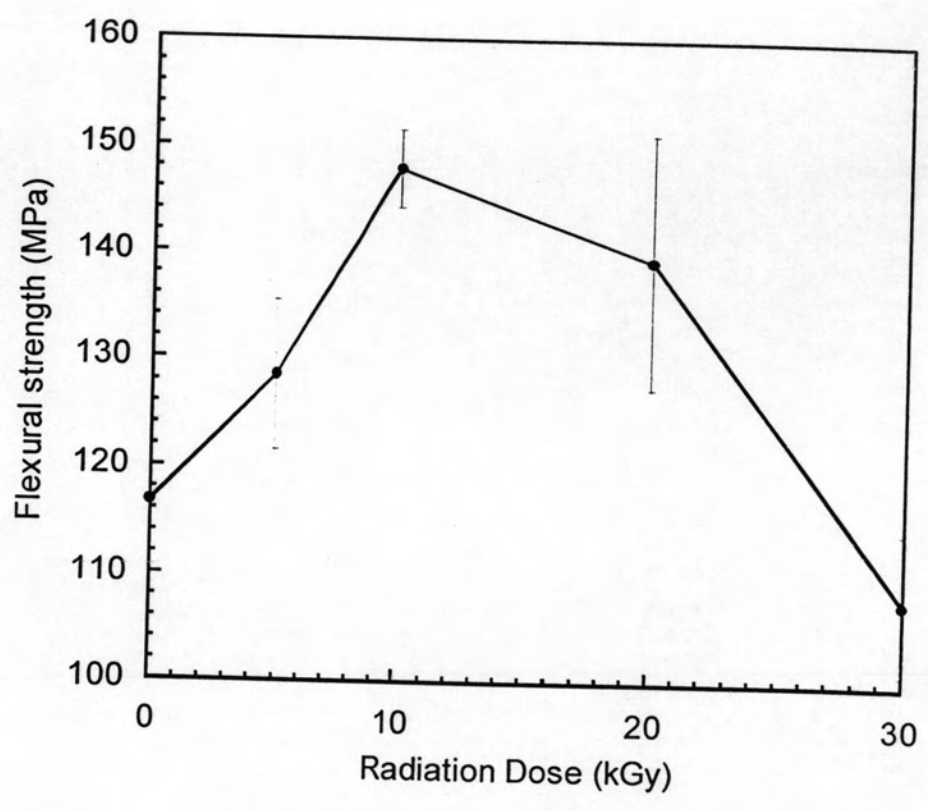


Figure 5.10: The flexural strength of rubber wood at various doses.

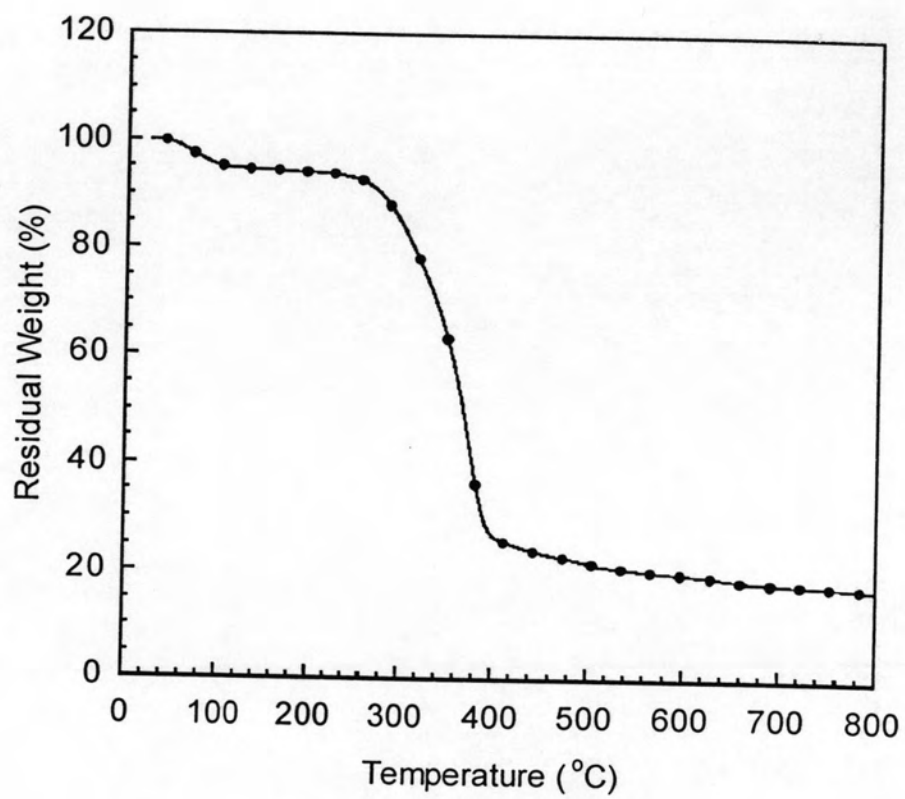


Figure 5.11: TGA thermogram of rubber wood.

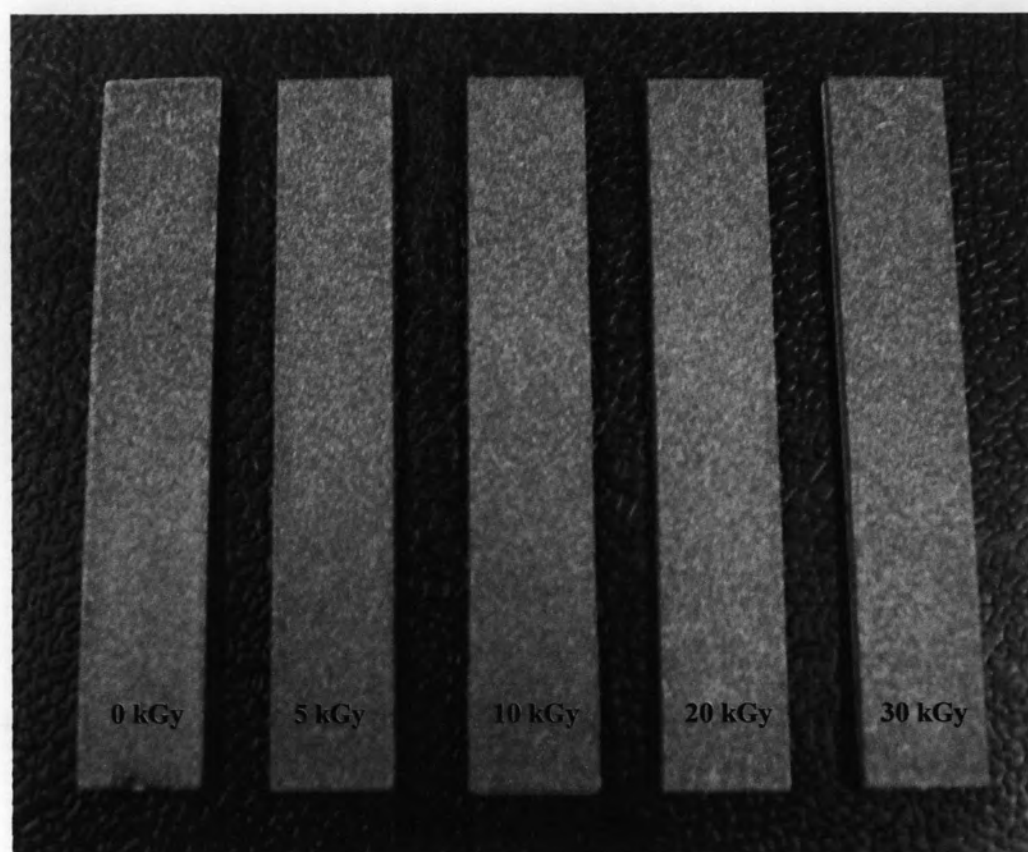


Figure 5.12: Effect of gamma irradiation on discoloration of PP woodflour composites.

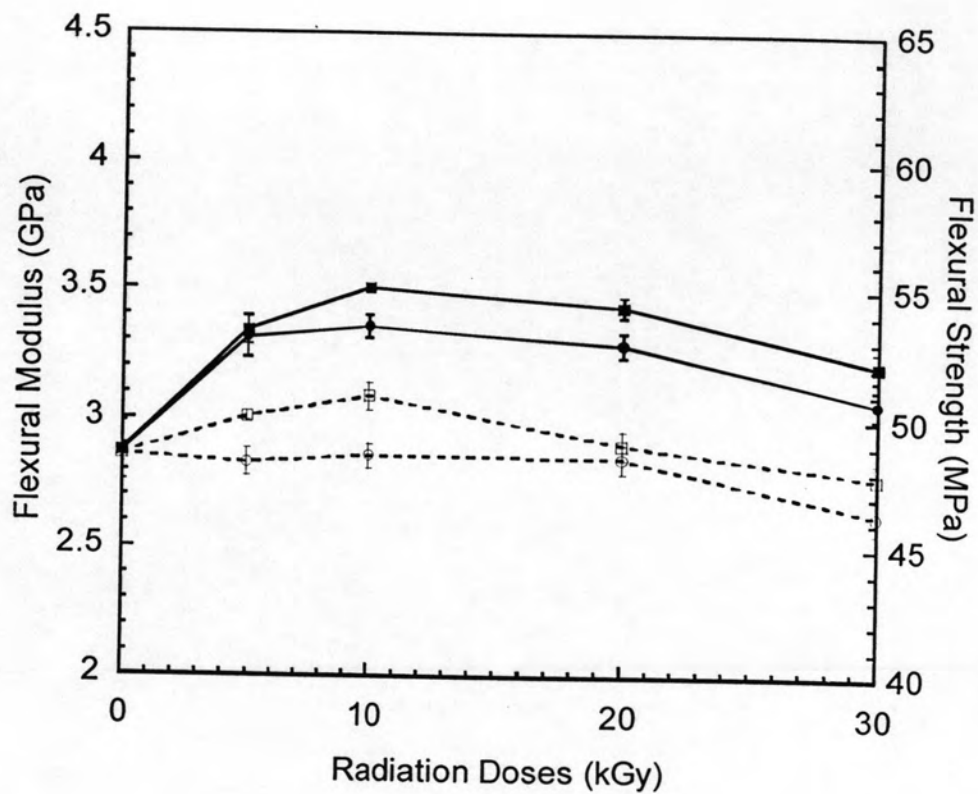


Figure 5.13: The flexural properties of polypropylene woodflour composites at various doses: (●) flexural modulus in air, (■) flexural modulus in nitrogen, (○) flexural strength in air, (□) flexural strength in nitrogen.

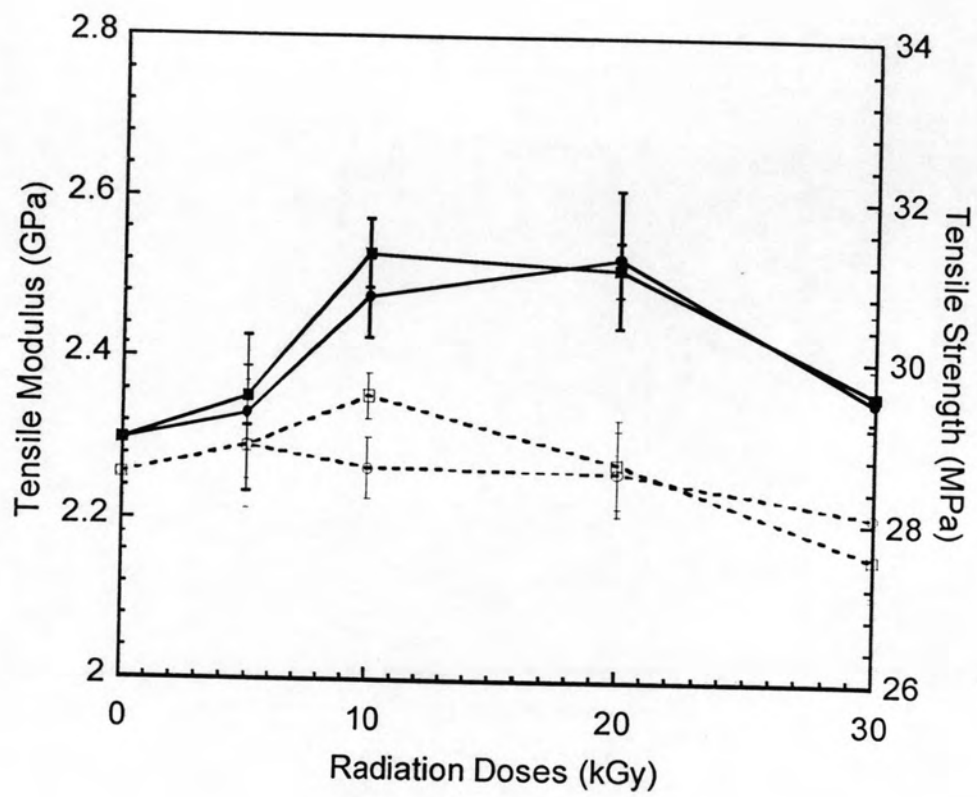


Figure 5.14: The tension properties of polypropylene woodflour composites at various doses: (●) tensile modulus in air, (■) tensile modulus in nitrogen, (○) tensile strength in air, (□) tensile strength in nitrogen.

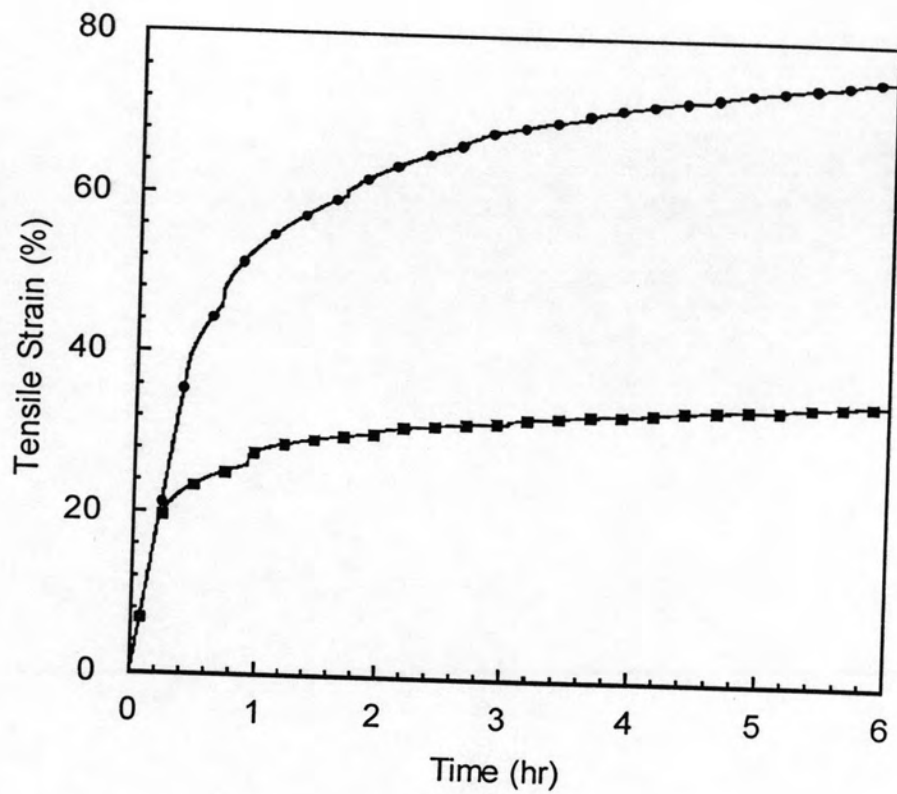


Figure 5.15: Creep behavior of polypropylene at various doses: (●) Polypropylene, (■) Polypropylene woodflour composite.



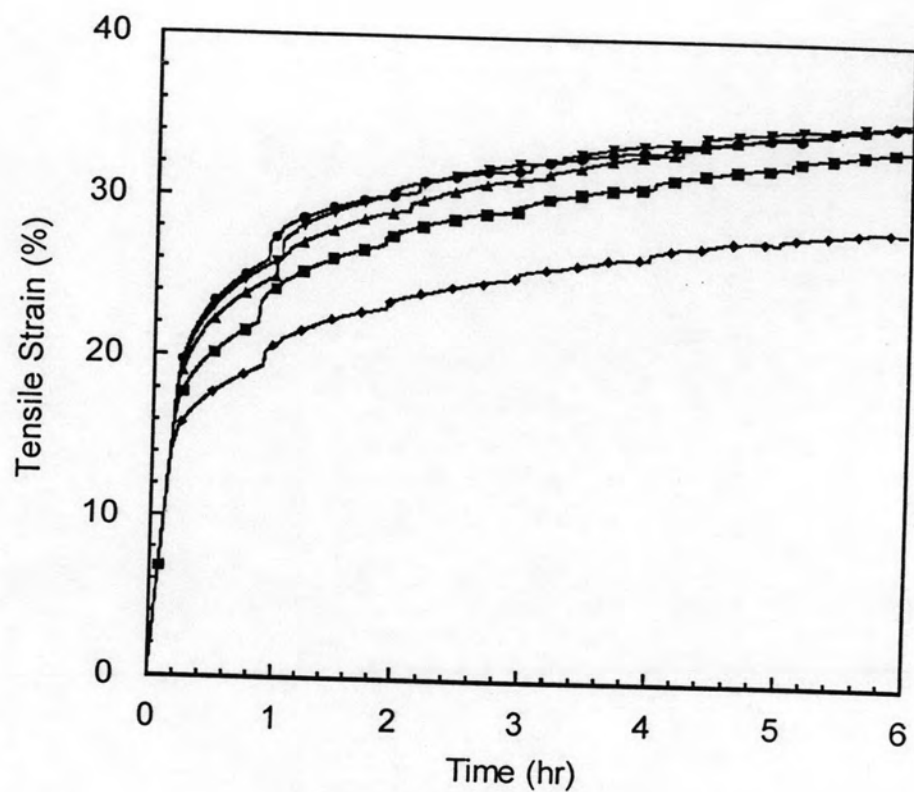


Figure 5.16: Creep behavior of polypropylene woodflour composites at various doses: (●) 0 kGy, (■) 5 kGy, (◆) 10 kGy, (▲) 20 kGy, (▼) 30 kGy.

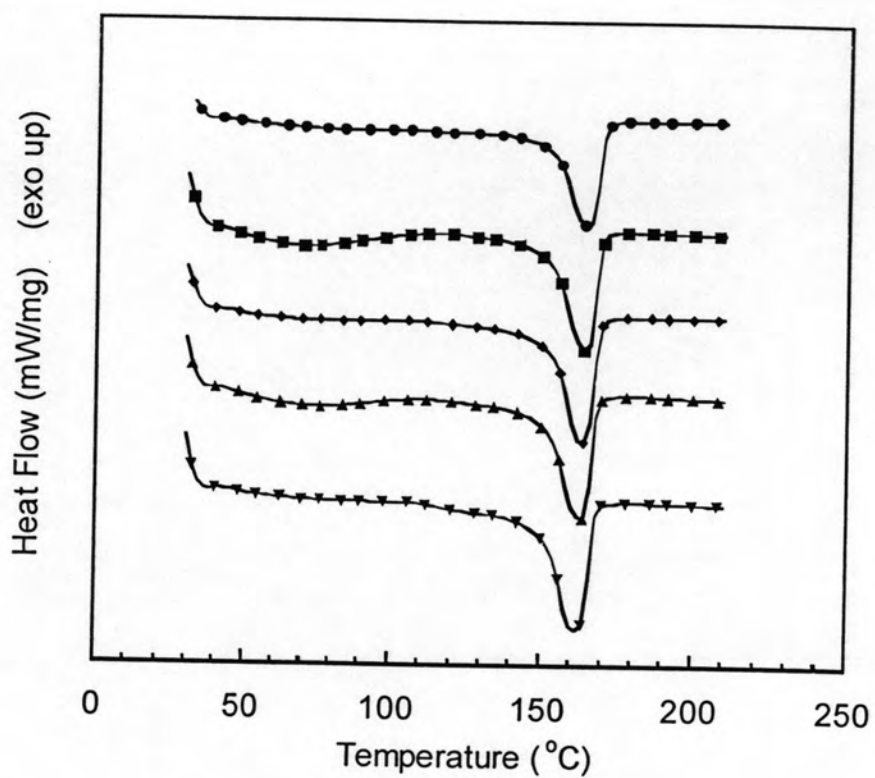


Figure 5.17: DSC thermograms of polypropylene woodflour composites at various doses: (●) 0 kGy, (■) 5 kGy, (◆) 10 kGy, (▲) 20 kGy, (▼) 30 kGy.

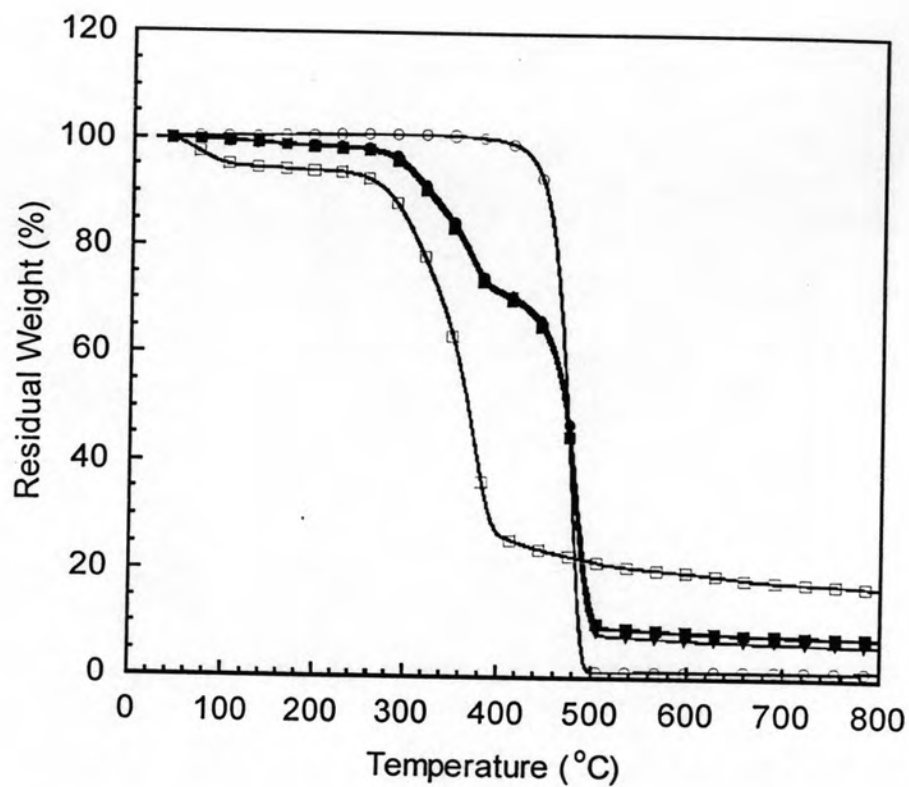
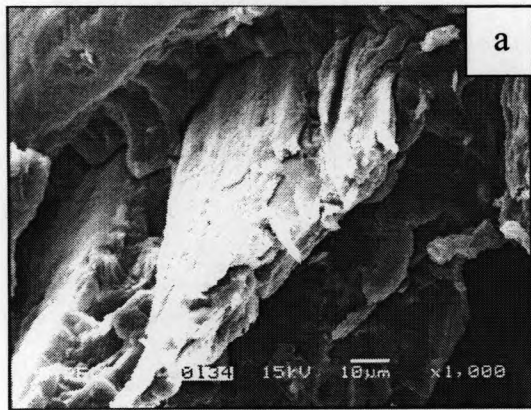
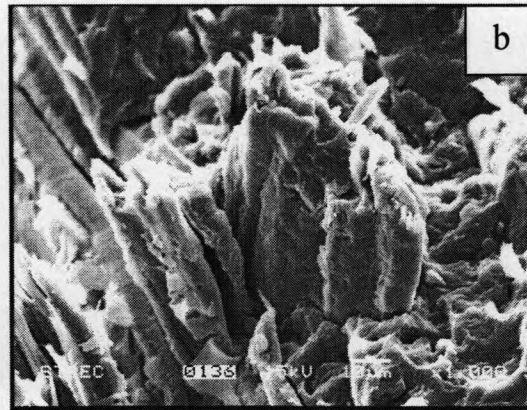


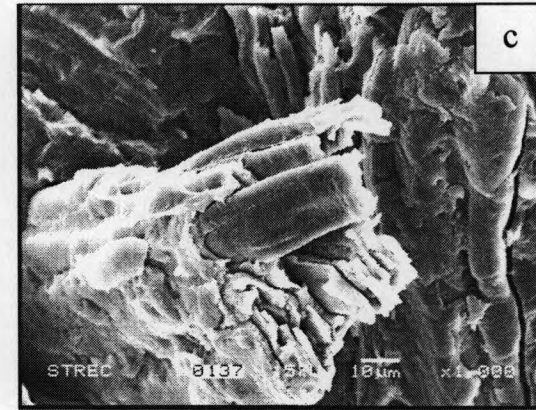
Figure 5.18: TGA thermograms of polypropylene, rubber wood and polypropylene woodflour composites at various doses: (o) Polypropylene, (□) Rubber wood, (●) 0 kGy, (■) 5 kGy, (◆) 10 kGy, (▲) 20 kGy, (▼) 30 kGy.



PP/woodflour composite 0 kGy



PP/woodflour composite 10 kGy



PP/woodflour composite 30 kGy

Figure 5.19: SEM micrographs of polypropylene woodflour composites at various radiation doses:
(a) 0 kGy, (b) 10 kGy, (c) 30 kGy.



Figure 5.20: Effect of gamma irradiation on discoloration of woodflour composites treated with maleic anhydride graft polypropylene.

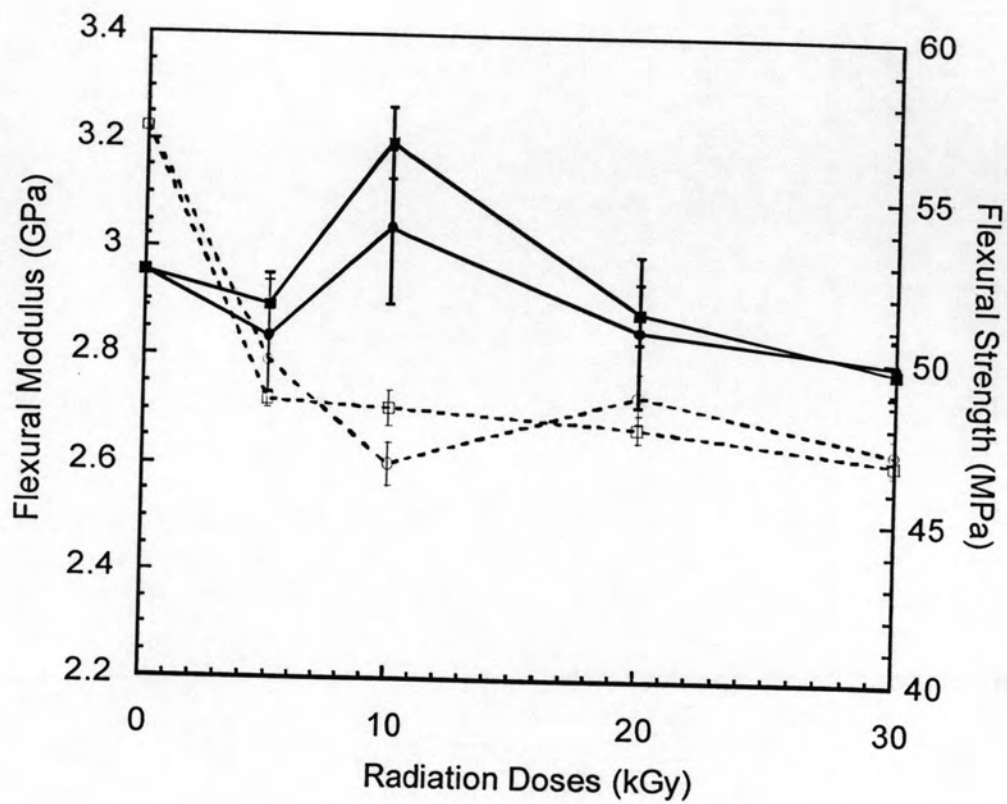


Figure 5.21: The flexural properties of polypropylene woodflour composites treated with meleic anhydride graft polypropylene at various doses: (●) flexural modulus in air, (■) flexural modulus in nitrogen, (○) flexural strength in air, (□) flexural strength in nitrogen.

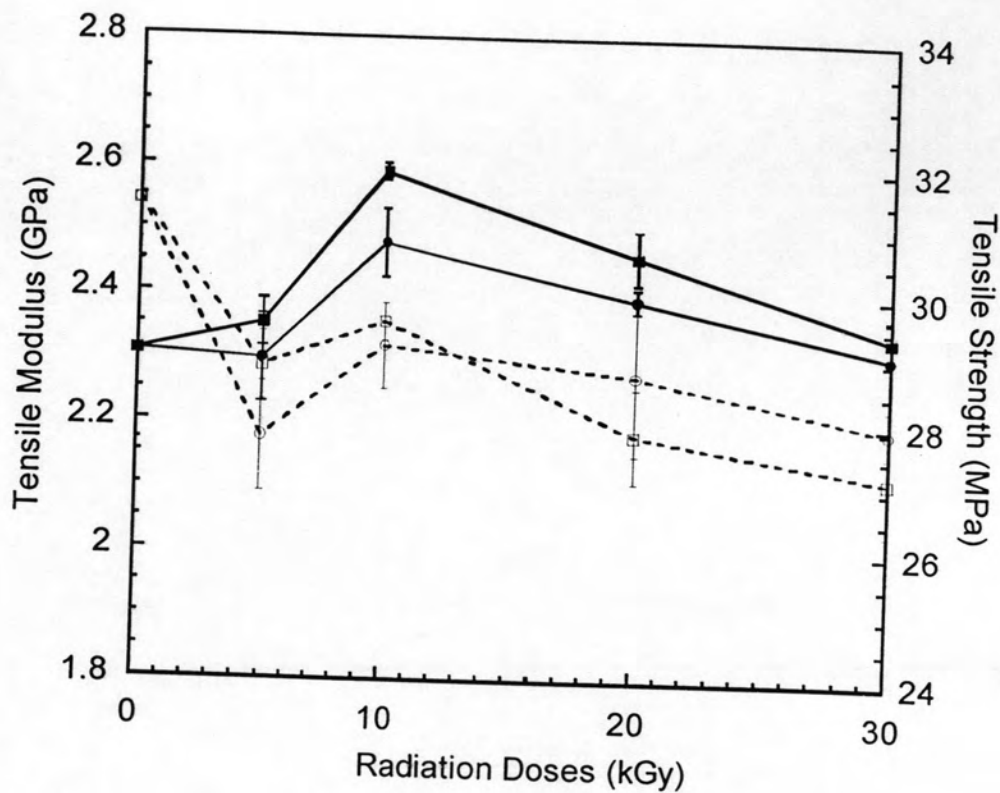


Figure 5.22: The tension properties of polypropylene woodflour composites treated with maleic anhydride graft polypropylene at various doses: (●) tensile modulus in air, (■) tensile modulus in nitrogen, (○) tensile strength in air, (□) tensile strength in nitrogen.

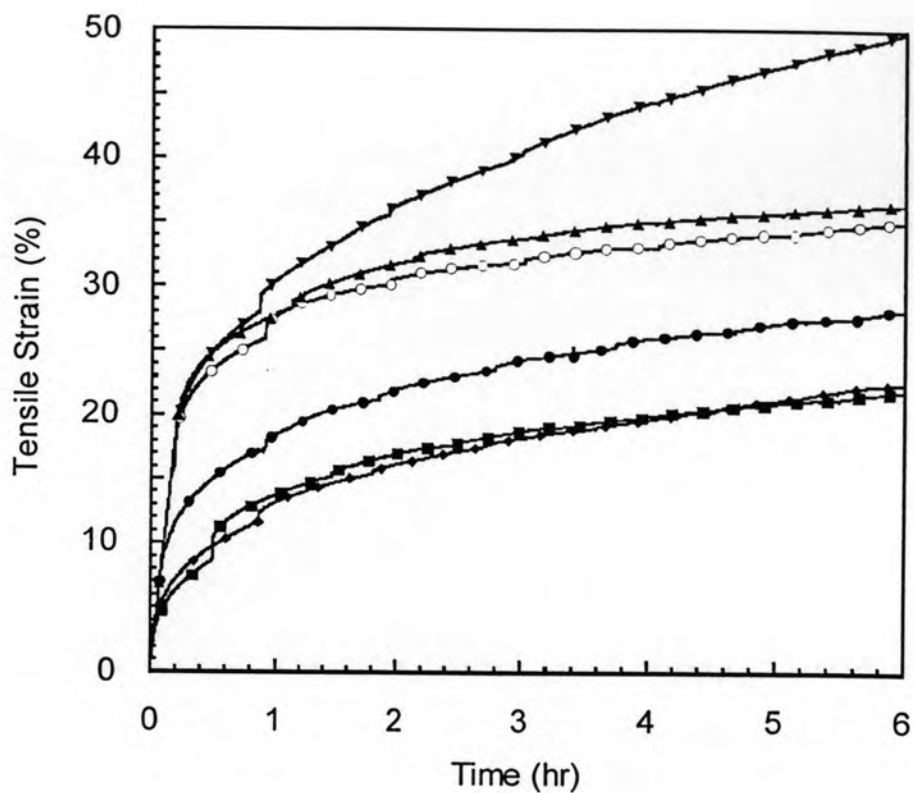
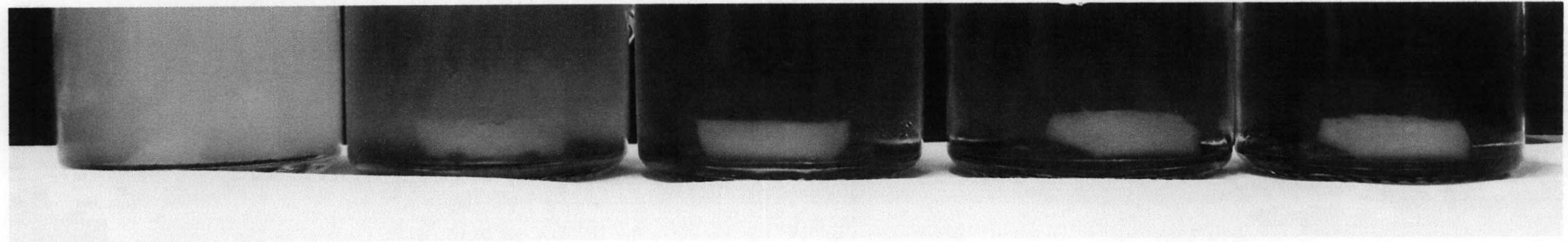


Figure 5.23: Creep behavior of polypropylene woodflour composites threated with maleic anhydride graft polypropylene at various doses: (○) PP/Wood, (●) PP/Wood/PP-g-MA 0 kGy, (■) PP/Wood/PP-g-MA 5 kGy, (◆) PP/Wood/PP-g-MA 10 kGy, (▲) PP/Wood/PP-g-MA 20 kGy, (▼) PP/Wood/PP-g-MA 30 kGy.



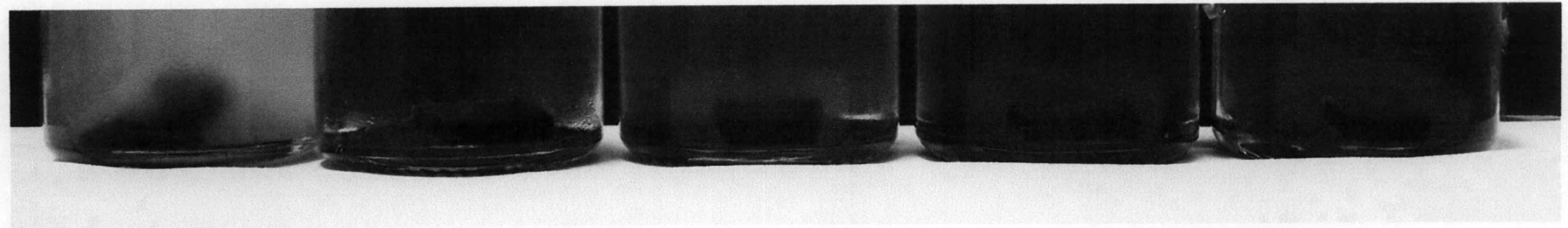
PP

PP 5 kGy

PP 10 kGy

PP 20 kGy

PP 30 kGy



PP/Wood 0 kGy

PP/Wood 5 kGy

PP/Wood 10 kGy

PP/Wood 20 kGy

PP/Wood 30 kGy

Figure 5.24: Xylene extraction of polypropylene and polypropylene wood composites at various doses.



PP/Wood/MAPP PP/Wood/MAPP 5 kGy PP/Wood/MAPP 10 kGy PP/Wood/MAPP 20 kGy PP/Wood/MAPP 30 kGy

Figure 5.25: Xylene extraction of polypropylene wood composites treated with maleic anhydride graft polypropylene at various doses

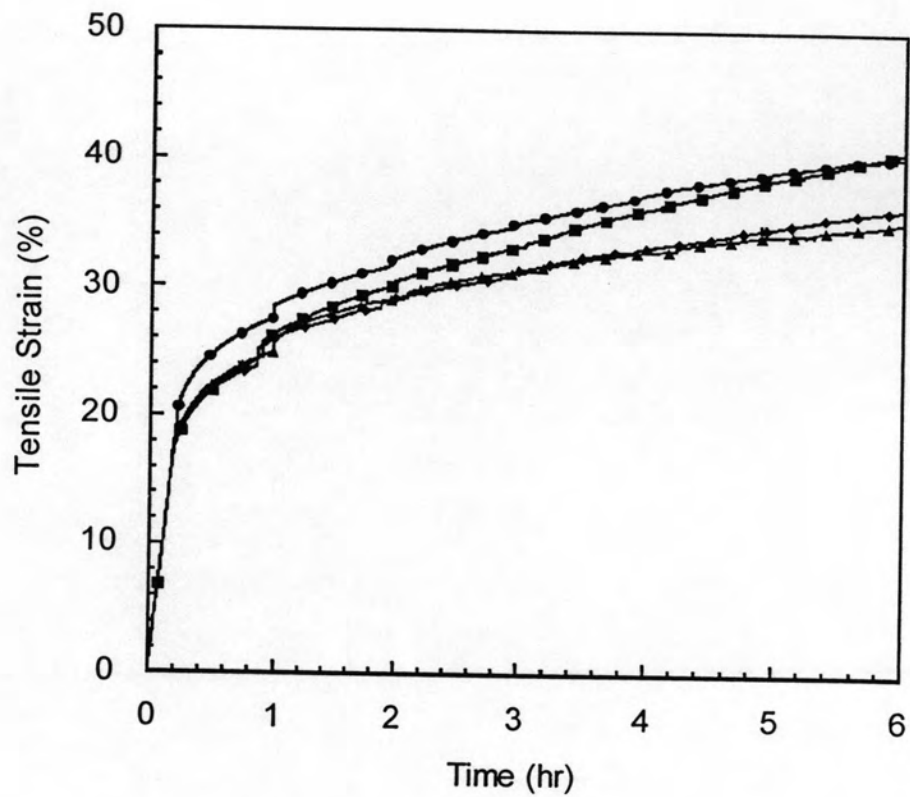


Figure 5.26: Creep behavior of polypropylene woodflour composites at various dose rates: (●) 0.0614 kGy/min, (■) 0.0850 kGy/min, (◆) 0.1515 kGy/min, (▲) 0.2112 kGy/min.