

COMPARISON OF THE OF MECHANICAL AND WEAR BEHAVIOURS OF DIFFERNT TYPES OF POLYETHYLENES AND THE EFFECT OF RADIATION CROSS-LINKING ON THESE BEHAVIOURS

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ABSTRACT

Different kinds of polyethylenes dominate the polymer field due to their wide range of applications and properties. They are sometimes cross-linked to improve their properties such as high temperature resistance and wear behavior. Radiation cross-linking using high energy electron beam or g-radiation constitutes one of the most convenient methods. This study explores the difference in mechanical and wear behavior of different polyethylenes, and compares the effect of radiation cross-linking and radiation dose on these properties. Three different types of polyethylenes: low density (LDPE), high density (HDPE), and ultra-high molecular weight (UHMWPE) were studied. Cross-linking was carried out by high energy electron beam at room temperature, with radiation dose ranging from 0-600 kGy.

The results show that the stress-strain curve of UHMWPE in unirradiated state is marked by extensive strain hardening resulting in excellent wear resistance. Unirradiated HDPE show extensive yielding and high strain to failure, with dry abrasive wear properties comparable to UHMWPE. Unirradiated LDPE on the other hand exhibit low strength and strain to failure, and comparatively high wear rate. UHMWPE has the highest cross-linking efficiency, while HDPE and LDPE show low cross-link densities. Cross-linking induces brittleness in the materials except in case of LDPE, and improves wear rate of LDPE and UHMWPE. However, the wear rate of HDPE increases with cross-linking.

KEY WORDS: Polyethylene, Cross-linking, Cross-link Density, Crystallinity, Mechanical Properties, Wear

1. INTRODUCTION

In general, polyethylenes possess good electrical insulation and chemical properties. The different classes of polyethylenes such as low density (LDPE), high density (HDPE), and ultra-high molecular weight (UHMWPE) are being used in a number of diverse applications. These can include house-wares and packaging through to cable insulation and high pressure piping¹⁻⁴. In most cases these polymers are required to show good mechanical properties at elevated temperatures, and good abrasive wear resistance^{5,6}. As a result, in recent years there has been a growing interest in enhancing the properties of these polymers by inducing cross-linking of the polymer chain through radiation cross-linking; and the effects of irradiation of polyethylenes have been studied extensively⁷⁻¹¹. In brief, this process uses high energy radiation in the form of high energy electrons or g-rays which can generate free radicals along the polymer chain by the abstraction of hydrogen atoms¹².

These free radicals then react to form a cross-linked network structure with a corresponding change in mechanical properties. Although the cross-linking process can be performed by other methods, radiation cross-linking is a convenient process which does not result in by-products, and produces varying degrees of cross-linking without reformulation. The commercial applications of cross-linked polyethylenes include high temperature applications like insulation on electrical wires and cables, hot water pipes and tubings, heat shrinkable products, and steam resistant food packaging¹³⁻¹⁶. Radiation cross-linked UHMWPE has also been widely used for making orthopaedic implants¹⁷⁻²¹.

In this study, the properties of 3 types of polyethylenes (LDPE, HDPE and UHMWPE) and the effect of radiation dosage on property changes have been investigated. The comparative data will assist in materials selection and in exploring the different ways to improve performance. The properties studied in-

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cluded; cross-link density (gel content) and crystallinity, tensile and shear behavior, and wear resistance under dry abrasive conditions. The research also compares and relates the effect of the degree of crystallinity and cross-link density induced within the polymer to subsequent changes in the mechanical and wear resistance properties of these polyethylenes.

2. MATERIALS AND METHODS

2.1 Materials

The additives present in polyethylene resin can interact with radiation, therefore, the polyethylenes selected for this study did not contain any additives (HDPE without additives was not available). The polyethylenes grades selected were: LDPE-EASTMAN 800A (Density ρ 0.918 g/cm³; Melt Flow Index MFI 1.7 g/10 min), HDPE-EASTMAN H6001-A (ρ 0.962 g/cm³; MFI 8 g/10 min), and UHMWPE-Hoechst Celanese GUR 1050 (ρ 0.930 g/cm³; MFI <0.01 g/10 min).

For LDPE and HDPE, a Morgan injection molding press was used for producing rectangular bars 9 mm thick, 25.4 mm wide and 100 mm long. The injection temperature was 218°C, injection pressure 0.4 MPa and clamp pressure 98 kN (for HDPE 133 kN). The mold temperature varied from 65-120°C. In the case of UHMWPE, commercially available bar-stock (ram extruded) material processed by WestLake Plastics, Lenni, PA, USA was used.

Irradiation was carried out using a 2.5 MeV Van de Graff generator at a dose rate of 25 kGy per pass (1 Mrad=10 kGy), at room temperature in air. Samples were given doses in the range of 100 to 600 kGy, in increments of 100 kGy.

2.2 Characterization

Swelling experiment was carried out according to ASTM test method D 2765 method C. Two samples (approximately 2-2.5 mg) of each material were dipped in xylene beaker, which was placed in a copper container, and heated on a hot plate. In the case of LDPE and HDPE specimens, xylene was heated to 110°C for 5 hours, while UHMWPE specimens were dipped in xylene at 130°C for 7 hours. The different temperatures were maintained due to the fact that control UHMWPE samples took more time to dissolve and cross-linked UHMWPE samples took more time to

equilibrate. The swollen gel was quickly transferred to a weighing bottle, covered and weighed. Samples were then dried in an oven at 85°C for 24 hours and weighed. The degree of swelling was calculated from the ratio of the weights of swollen and dry samples. The cross-link density (ν_c) in moles per unit volume was calculated from the Flory's swelling equation given below:

$$\nu_c = - \frac{\ln(1 - q^{-1}) + q^{-1} + \chi q^{-2}}{\bar{V}_1 q^{-1/3}} \quad (1)$$

where q is the swell ratio, \bar{V}_1 is the partial volume of the diluent (xylene) and is 136 ml/mole, χ is the Flory-Huggins interaction parameter and is defined by the following equation: $\chi=0.33+0.55/q$. Molecular weight between cross-links (M_c) is calculated from the following:

$$M_c = \frac{\rho}{\nu_c} \quad (2)$$

where ρ , the density, is equal to the density of the amorphous polyethylene (0.8621 g/cm³).

Differential scanning calorimetry (DSC) of the samples was carried out on a Perkin-Elmer DSC 7a model to determine the degree of crystallinity and melting points of the different materials. The heating rate was 10°C/min. Three different samples: un-irradiated (control), and 200 kGy and 600 kGy radiated samples of each series were tested. Results were also used to ascertain any degradation taking place in the samples. The heat of fusion of the materials was compared with the heat of fusion of 100% crystalline polyethylene, taken as 289 J/g.

2.3 Mechanical Testing

The LDPE and HDPE specimens used for mechanical tests were processed from extruded sheet supplied by Commercial Plastics, Somerville, MA, USA (their cross-link densities were measured in separate experiments), while UHMWPE specimens were machined from bar-stock material mentioned earlier. Control and 200 kGy radiated samples were tested in each case. The tensile test was performed on standard ASTM D638 dog-bone specimens. The dimensions of the narrow section were; length (or gage length) 9.53 mm, width 3.18 mm and thickness 3 mm. The overall length and width of the specimen were 63.5 mm and 9.53 mm. The radius of the fillet was 8 mm. An Instron 5567 testing machine was used and the force was measured by a 30 kN load cell. The cross-head speed was 10 mm/min. The deformation was obtained directly from the motion of the crosshead. Four speci-

mens were tested in each case. Zero slope yield point was used to calculate the yield strength and strain.

A shear test method based on the designs of G'Sell *et al.*²² and Lin and Argon²³ was devised to measure the shear stress-strain behavior²⁴. Figure 1 shows a schematic diagram of the setup and the specimen. It produced simple shear in the center slot of reduced thickness, with no moments applied. The specimen was sandwiched between two steel plates. The specimen and the steel plates were held together by screws. An Instron machine was used at a cross-head speed of 1.27 mm/min, producing a shear strain rate of 0.019 sec⁻¹. Deformation was measured from the motion of the crosshead. Shear stress (τ) and strain (γ) were calculated from the following equations:

$$\tau = \frac{P}{\ell t} \text{ and } \gamma = \frac{x}{h} \quad (3)$$

Where P is the load measured by the load cell and x is the displacement of the crosshead. Length (ℓ), thickness (t) and width (h) of the center slot were 20.3, 1.1 and 0.6-1 mm, respectively.

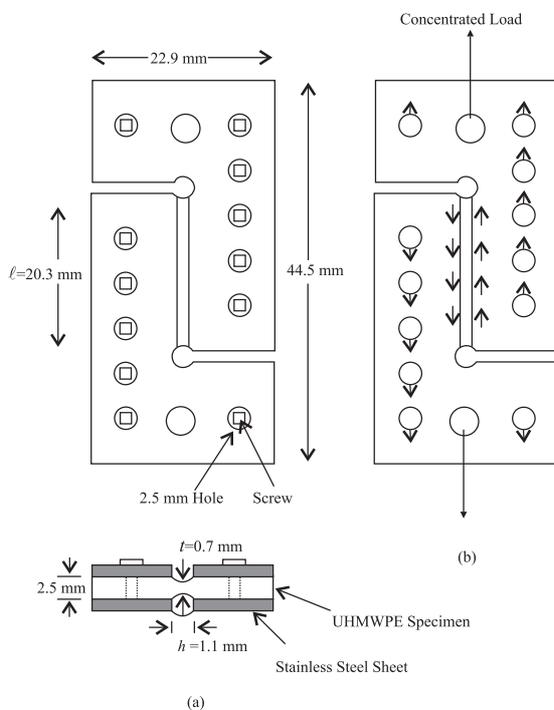


Figure 1: (a) Schematic diagram of the simple shear setup and the specimen. (b) The loading diagram.

2.4 Wear Test

The wear rate of the control and 200 kGy radiated polymer samples was compared using a pin-on-disc wear testing machine under dry abrasive conditions. The polymer was machined to a cylindrical shape to form the “pin” which had a diameter of 5 mm and length 10 mm. The pin was held against a SiC abrasive paper (600 grain size) attached to the disc using a load of 1 Kg. The test was performed with a sliding speed of 1 m/s for a total sliding distance of 730 m. A new SiC paper was used for each polymer sample tested. No lubricant was used and no attempt was made to clean the abrasive paper from transferred debris. In some tests the pin suffered from extreme wear and a shorter sliding distance had to be used. The wear rate was determined by recording the change in pin weight as a function of sliding distance.

3. RESULTS

Figure 2 shows the effect of radiation dose on the cross-link density of the three different types of polyethylenes measured by the swelling experiment. The results show that HDPE exhibits a linear increase in cross-link density with radiation dose ($y=14.0x-6.89$; $R^2=0.9953$). The cross-link density of LDPE and UHMWPE increases with the radiation dose and appears to reach a saturation limit around 300 kGy. Highest cross-link density is achieved in UHMWPE, followed by LDPE and HDPE.

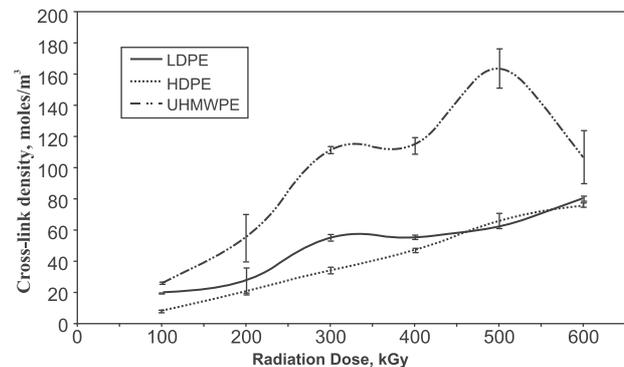


Figure 2: Effect of radiation dose on the cross-link density of the different types of polyethylenes.

The degree of crystallinity and onset melting point measured by DSC analysis are shown in Table 1. The comparison of the degree of crystallinity of un-irradiated samples shows that percent crystallinity of LDPE is 34%, while HDPE has the highest crystallinity 67%. UHMWPE lies in between at about 50% crystallinity. Cross-linking does not produce appre-

ciable change in the degree of crystallinity and melting points, as it remain constant with radiation dose in most of the cases. The small variation can be attributed to measurement error, except in the case of UHMWPE where appreciable increase in crystallinity is observed due to possible degradation in molecular weight.

Table 1: Degree of crystallinity (%) and the onset melting point (°C) of the control and cross-linked polyethylenes measured by the DSC analysis.

Sample ID	Degree of Crystallinity, %	Onset Melting Temperature, °C
LDPE Control	33.7	104.4
LDPE 200 kGy	32.1	100.6
LDPE 600 kGy	34.5	98.2
HDPE Control	67.3	128.2
HDPE 200 kGy	68.8	132.3
HDPE 600 kGy	67.2	128.3
UHMWPE Control	49.7	130.0
UHMWPE 200 kGy	60.7	132.9
UHMWPE 600 kGy	67.7	133.8

It may be mentioned that the real time aging effects such as increase in crystallinity due to oxidation could not be controlled in this study. In addition, presence of antioxidant in some of these polymers can interfere with the radiation crosslinking.

Figure 3 shows comparison of the typical tensile stress-strain curves of control LDPE, HDPE, and UHMWPE, while Figure 4 shows comparative tensile stress-strain curves for control and 200 kGy radiated samples of these materials. In most samples, a neck forms on yielding, which yields further and expand to the entire gage length. The Control HDPE shows very high strain to failure as compared to LDPE and UHMWPE which show similar values. Its zero slope yield point is also the highest closely followed by UHMWPE, while LDPE has very low yield stress. Extensive strain hardening is a marked feature of UHMWPE stress-strain curve which lead to very high failure stresses. The results of the radiated samples show that irradiation drastically reduces the strain to failure of both HDPE and UHMWPE samples, while the yield strength (on zero slope yield point) increases to some extent. LDPE on the other hand show entirely different behavior, the strain to failure and toughness increases with cross-linking. Table 2 shows the tensile yield and fracture properties calculated from

the tensile tests of different materials, and confirms the above observations. The table also reports the cross-link densities of the samples used for mechanical tests.

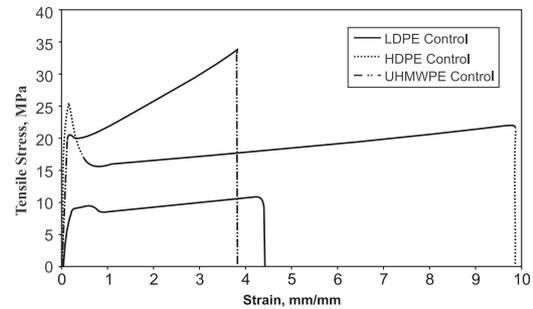


Figure 3: Tensile stress-strain curves of un-irradiated (Control) LDPE, HDPE and UHMWPE samples.

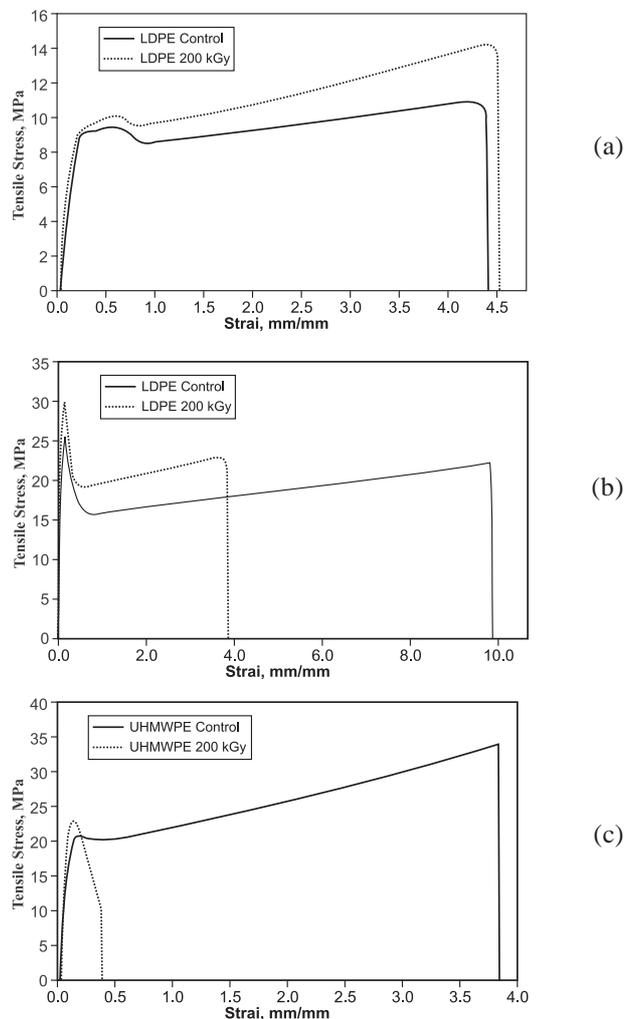


Figure 4: Comparison of the tensile stress-strain curves of un-irradiated (Control) and 200 kGy radiated samples of (a) LDPE, (b) HDPE and (c) UHMWPE.

Table 2: The tensile yield stress (σ_{Ty}) and strain (ϵ_{Ty}), and tensile failure stress (σ_{Tf}) and strain (ϵ_{Tf}) of the tested materials. Degrees of cross-linking of the radiated materials are also given. The yield properties are measured at the zero slope yield point. The standard deviation values are reported in the brackets.

Sample ID	Degree of Cross-linking, moles/m ³	σ_{Ty} MPa	ϵ_{Ty} mm/mm	σ_{Tf} MPa	ϵ_{Tf} mm/mm
LDPE Control	-	9.44 (± 0.04)	0.533 (± 0.036)	9.71 (± 0.59)	4.167 (± 0.551)
LDPE 200 kGy	21.6	10.06 (± 0.09)	0.581 (± 0.054)	14.20 (± 1.26)	4.871 (± 0.607)
HDPE Control	-	25.94 (± 0.61)	0.149 (± 0.010)	21.07 (± 1.30)	9.383 (± 1.504)
HDPE 200 kGy	34.2	29.70 (± 0.36)	0.125 (± 0.031)	21.87 (± 1.50)	3.325 (± 0.820)
UHMWPE Control	-	20.31 (± 0.30)	0.184 (± 0.010)	33.09 (± 3.49)	3.948 (± 0.803)
UHMWPE 200 kGy	55.2	23.80 (± 0.47)	0.140 (± 0.000)	10.44 (± 1.09)	0.376 (± 0.017)

Representative shear stress-strain curves for the different un-irradiated polyethylenes are shown in Figure 5. No zero slope yield point is present in UHMWPE; yielding is shown by a change in slope in the initial part of the curve followed by extensive strain hardening until the specimen fractures. With HDPE a zero slope yield point is present and the material yields until it fractures; while with the LDPE no zero slope yield point is observed. Some strain hardening can be seen in the LDPE sample but the slope is very small. Radiation cross-linking produces degradation in the shear behavior of HDPE and UHMWPE while improves the toughness of LDPE; an observation similar to the tensile test (Figure 6). The extent of decrease in strain to failure of HDPE and UHMWPE samples is less significant than tensile test, while the improvement in strain to failure of LDPE is more pronounced. Irradiation produces a small increase in shear yield strength of HDPE and UHMWPE.

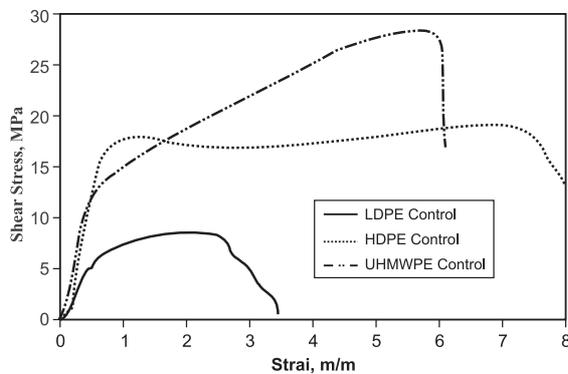


Figure 5: Shear stress-strain curves of un-irradiated (Control) LDPE, HDPE and UHMWPE samples

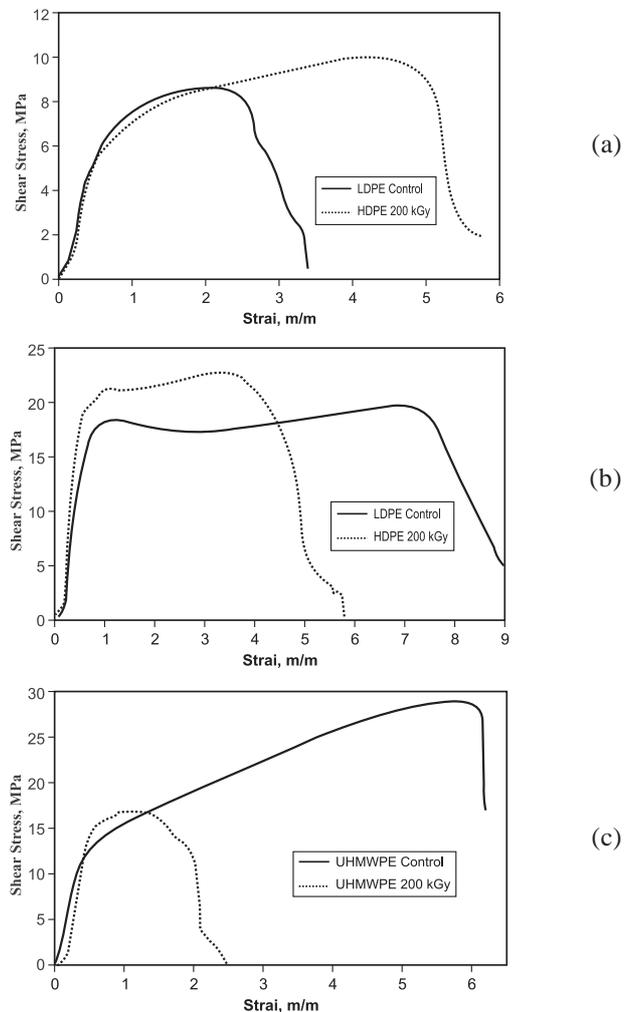


Figure 6: Comparison of the shear stress-strain curves of un-irradiated (Control) and 200 kGy radiated samples of (a) LDPE, (b) HDPE and (c) UHMWPE.

The comparison of the wear behavior of the control un-irradiated samples of 3 polyethylenes is presented in Figure 7. LDPE shows distinctly high wear rate in contrast to HDPE and UHMWPE. The transfer of material on the abrasive paper was also different in the two categories: in LDPE, fine flake-like wear debris was present, but not as a continuous transfer film; the pin exhibited extreme wear and lip formation. In the case of HDPE and UHMWPE, smaller size loose debris was produced leading to a smooth and continuous transfer film on the abrasive surface. The transfer film plays a significant role in reducing further wear of the polymer surface, and provides a counter-surface with low coefficient of friction. Lip formation was not observed in these materials. The comparison of 200 kGy irradiated specimen with the control also exhibit two distinct behaviors (Figure 8). In LDPE and UHMWPE, wear resistance improved with irradiation, while in HDPE the wear resistance degraded somewhat with irradiation. The most wear resistant material in this study was radiated UHMWPE, and on the other extreme the least wear resistant material was unirradiated control LDPE.

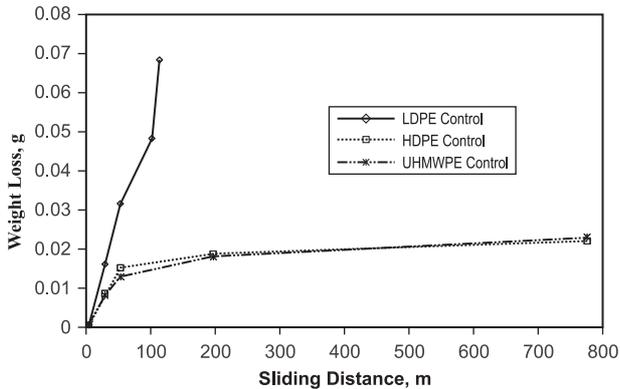


Figure 7: Weight loss (g) versus sliding distance (m) curves for un-irradiated (Control) polyethylenes measured from a pin-on-disc wear test under dry abrasive conditions.

4. DISCUSSION

To explain the differences in behavior of the different polyethylenes, it is important to understand the skeletal structure of these materials. LDPE has a highly branched structure with crystallinity ranging from 30-40%, as opposed to HDPE which has a linear structure and crystallinity as high as 75%^{4,25}. As its

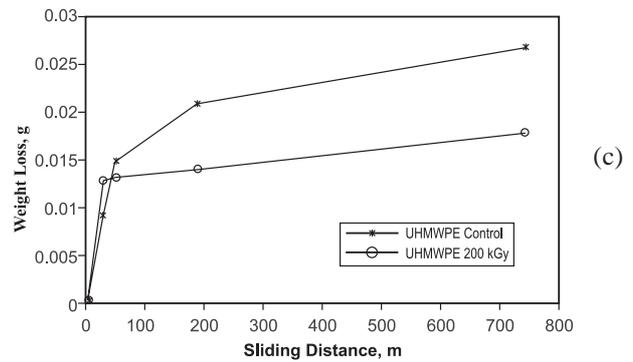
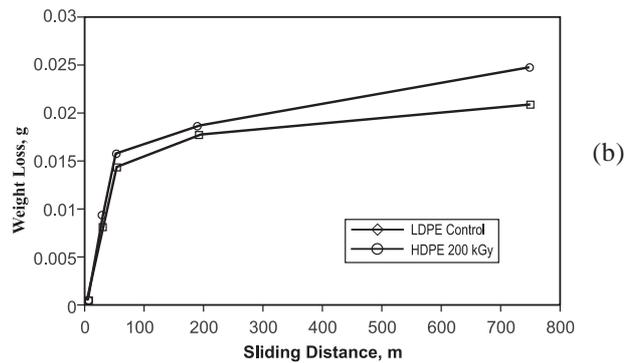
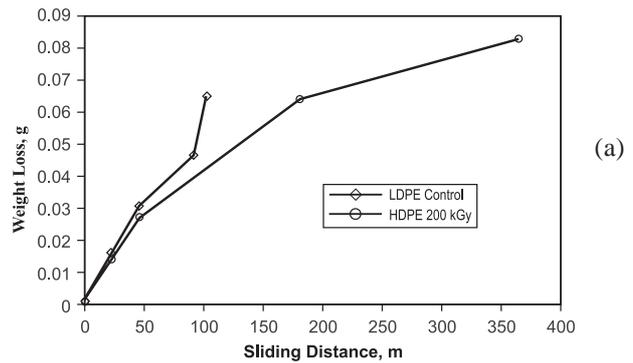


Figure 8: Comparison of the wear behavior of un-irradiated (Control) and 200 kGy radiated samples of (a) LDPE, (b) HDPE and (c) UHMWPE.

name suggest UHMWPE has very high molecular weight linear molecules, which leads to lower crystallinity (typically 45 %²⁶) and higher entanglement densities. The differences in behavior are due to the relative amounts of crystallinity and entanglement density, with chain branches and higher molecular weight promoting higher entanglement density and lower crystallinity.

The lower cross-link density achieved in HDPE is due to the fact that cross-linking mainly occurs in amorphous regions which are very small in HDPE. The cross-link densities achieved in UHMWPE are very high as compared to other materials due to its higher molecular weight leading to larger entanglement densities and larger potential cross-linking sites.

The saturation may represent a complete cross-link network and fulfilment of all the entanglement and branch sites available for cross-linking. LDPE show similar behavior but lower cross-link densities; this is due to smaller average molecular weight of LDPE as evident by its large melt flow index (MFI); a smaller MFI indicates a higher molecular weight. As compared to HDPE, LDPE has higher cross-link density at similar radiation dose as also reported in earlier studies¹⁵. This is mainly due to the lower crystallinity and higher branch content of LDPE as compared to HDPE.

The DSC analysis confirms that the degree of crystallinity is in range of that expected from the literature. The crystallinity and melting point did not change appreciably with increasing cross-link density which suggests the absence of degradation in molecular weight by oxidation and/or radiation damage. Radiation-induced chain scissioning or oxidative damage may increase the number of small chains and any such change may induce recrystallization, thus increasing the degree of crystallinity. The exception is UHMWPE where appreciably high increase in crystallinity is observed due to degradation in molecular weight. The reason is that free radicals produced during radiation exposure in UHMWPE can survive for a long time due to diminished molecular motion because of its high molecular weight; as oxygen diffuses in the material the radicals are consumed leading to further degradation in molecular weight. The lower molecular weight fragments produced are able to reorganise and crystallize more readily¹¹. Since the samples were analysed after two to three years of storage enough time was available for the diffusion of oxygen and subsequent degradation. The technique employed to avoid this problem is to melt anneal UHMWPE after radiation exposure to extinguish any free radicals present and add antioxidants such as vitamin E^{27,28}.

The results of both tensile and shear tests are comparable to a large extent. Control LDPE in both

tests showed appreciably lower yield stress, strain to failure, and small strain hardening slope. Control HDPE showed extensive yielding with negligible strain hardening slope and very high strain to failure; this is in agreement with previously reported results,^{22,29}. While control UHMWPE show extensive strain hardening causing very high failure stresses. Cross-linking and long-term oxidation both are expected to decrease strain to failure in HDPE and UHMWPE; the extent of this degradation in mechanical properties is however less significant in the shear test results. On the other hand, radiated LDPE showed an increase in strain to failure and toughness; this inconsistent behavior needs further study and evaluation.

The difference in mechanical behavior of the three types of polyethylenes studied herein is believed to be due to the relative amount of entanglement density and crystallinity. In HDPE the crystallinity is very high and the number of entanglements is very small. Thus, the deformation mechanisms are dominated by the crystalline plasticity; the amorphous phase is too small and not entangled to produce any strain hardening. In UHMWPE extensive strain hardening is observed due to the lower crystallinity and a larger numbers of molecular entanglements present because of its extensively high molecular weight. The amorphous, highly entangled phase, will contribute to the strain hardening with the crystallites also acting as entanglement sites enhancing the strain hardening behavior. Limited strain hardening observed in LDPE is due to its entangled structure produced by chain branching. However, the comparatively smaller strain hardening modulus is due to smaller entanglement density as compared to UHMWPE.

The comparison of the wear behavior of control polyethylenes show that LDPE experiences very high wear rate as compared to HDPE and UHMWPE, which show comparable and very small wear rate. This observation can be explained on the basis of the smooth transfer film produced in the two later cases leading to reduced friction coefficient and reduction of further wear. The production of this continuous transfer film can be further explained on the basis of high strain to failure and toughness observed in HDPE and UHMWPE as shown by their stress-strain curves particularly from the shear test. Marked difference in behavior is observed in these materials as compared to LDPE leading to very high abrasion resistance.

Cross-linking improves the wear resistance of LDPE and UHMWPE, and degrades it somewhat in HDPE. Wear mechanisms of polyethylenes can be divided in the following categories; adhesive, abrasive, and surface fatigue wear. Adhesive and surface fatigue wear involves the removal of polymer by the harder counterface asperities after many interactions, while abrasive wear involves the ploughing of polymer by the surface asperities of a harder material and is associated with rough surfaces^{30,31}. Wear test used in this study is likely to produce abrasive wear due to absence of any lubricant and presence of an abrasive counter surface. The decrease in wear rate of radiated LDPE and UHMWPE is due to the formation of extensive cross-link network structure in these cases. This network structure is produced by the cross-linking of large number of entanglements produced by the very high molecular weight of UHMWPE and by the branched structure of LDPE. The cross-links in the network structure will obstruct the formation of wear debris by resisting the uncoiling of the physical entanglement; leading to an improved wear behavior. The toughness increase in LDPE due to cross-linking will also assist in increasing the wear resistance. Due to the linear molecular structure of HDPE the network formation will not be extensive. This coupled with degradation in its mechanical properties such as toughness due to cross-linking will lead to decreased wear resistance with radiation. Similar observations about wear under dry conditions have also been reported in previous studies³².

5. CONCLUSIONS

The results provide a comparison of the properties of different types of un-irradiated and radiated polyethylenes. It shows that UHMWPE has the highest cross-linking efficiency, followed by LDPE and HDPE, respectively. The cross-link density of UHMWPE and LDPE has approached a saturation limit at 300 kGy, while the cross-link density of HDPE samples increases linearly with radiation dose. The degree of crystallinity does not change appreciably with irradiation since it is carried out at room temperature, except in case of UHMWPE where some degradation in molecular weight due to diffusion of oxygen causes an increase in crystallinity. The mechanical behavior of the different materials as observed by the tensile and shear tests is significantly different. Un-irradiated UHMWPE exhibit significant strain harden-

ing, while un-irradiated HDPE show extensive yielding with insignificant strain hardening and very high strain to failure. Control LDPE on the other hand show very low strength and strain to failure but some strain hardening. The different stress-strain behavior observed can be explained by the different ratios of entanglement density and crystallinity present in the materials; with entanglement density determining the strain hardening modulus. Cross-linking sharply decreases the strain to failure and toughness of HDPE and UHMWPE with some increase in their yield strength. However, in LDPE the strain to failure and toughness increases after cross-linking.

Wear test results show that control LDPE exhibits the highest wear rate. In contrast HDPE and UHMWPE exhibit very low wear rate due the formation of a continuous transfer film. Radiation cross-linking decreases the wear rate in LDPE and UHMWPE, while increases the wear rate to some extent in HDPE. Therefore, cross-linking is only a promising method to improve the wear properties of the former two materials. This effect is due to the formation of extensive network structure aided by the high entanglement density present in LDPE and UHMWPE. The toughness increase in LDPE due to cross-linking is also a source of this improvement in wear properties. Under dry abrasive conditions the most wear resistant material investigated in this study is radiated UHMWPE.

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