Effect of Gamma irradiation on mechanical behavior of EPDM rubber-recycled news print microfibers composites

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Abstract

The waste newsprint paperswere collected from local market and submitted to chemical pulping in a kneader with 2M NaOH. The fiber after getting rid of water, treated again using 2M HCL solution for the same time period. Then the news print pulped microfibers (NPMF) batch obtained was mixed with Ethylene propylene diene monomer rubber (EPDM) on roll mill with differnet contents ranged from 5 to 50phr (part per hundred part of rubber). The prepared composites subjected to gamma irradiation at different doses from 20 up to 100kGy. The characterization of the microfibers carried out by different tools like scanning electron microscopy (SEM), X-ray diffraction (XRD),Forrier Transform Infrared spectra (FTIR) and Thermogravimetric analysis (TGA). The mechanical properties of prepared composites like tensile strength (Ts), Elongation at break (E_b%), tensile modulus(M_{100}) , tensile toughness and crosslink density (Cd) were measured as a function of fiber contents and irrdaition dose. The results indicated the Ts was increased with increasing fiber content up to 10phr and incresed with increasing irradiation dose up to 40kGy, while E_b% decreased by fiber content and irradiation dose. M_{100} and Cd were incresed with invreasing fiber content up 50phr fiber and increased by irradiation dose up to 60kGy. The results concluded that toughness values of EPDM/ news print fiber composite reach maximum using 10phr fiber concentration and 60kGy irradiation dose.

Keywords:waste newsprint microfibers (NPMF)- EPDM/news print fiber composites Chemical repulping - Thermogravimetric analysis (TGA) - X-ray difrraction (XRD) - FTIR analysis– Mechanical properties.

1.Introduction

A great deal of interest has been generated in the use of natural fibers as reinforcing fillers in thermoplastic or elastomer composites. The low densities of lignocellulosic fibers coupled with their low cost makes these fibers potentially attractive for non-structural composite applications[**Allen P.W., 1972**].The intra and inter-chain hydrogen bonding network makes cellulose a relative stable polymer, and gives the cellulose fibrils high axial stiffness [**Moon R.J.,et al., 2011**]. The high cohesive energy ensuing from these physico-chemical interactions explains why cellulose does not possess a liquid state [**Gandini A., 2011**] and these cellulose fibrils are the main reinforcement phase in trees and plants. Terpolymerization of ethylene, propylene and a non-conjugated diene yields ethylene-propylene diene-monomer (EPDM) rubber with a saturated polymer backbone and residual unsaturation in the side groups. As a result, EPDM rubber has a superior resistance against oxygen, ozone, heat and irradiation over poly-diene rubbers, such as natural rubber (NR), butyl rubber (BR) and styrene-butyl rubber (SBR). This makes EPDM the elastomer of choice for outdoor and elevated-temperature applications, such as automotive profiles, window gaskets, roof sheeting, wastewater seals, electrical cable & wires and V belts. As other elastomers, EPDM had to be cross-linked to achieve its optimum performance in terms of elasticity, tensile, tear strength and solvent resistance. Introduction of unsaturation via incorporation of the diene monomer enables sulfur vulcanization (80% of EPDM applications) and enhances the peroxide curing efficiency (15%). The obvious advantage of

peroxideas well as irradiation curing over sulfur vulcanization is the formation of thermo-stable C-C bonds instead of thermo-labile S-S bonds. Peroxide cure, therefore, allows the full exploitation of the excellent heat resistance of EPDM[**Treloar L. R. G., 1975**].

Compared with the conventional chemical processes used for crosslinking of rubber, radiation crosslinking has advantages over them for being faster, more versatile, for bringing about uniform crosslinking, for consuming less energy and finally for the inherently waste-free of the technology,[**Bly, J., 1983**]. It is well known that the exposure of crosslinking type polymers to radiation provides improved stability and mechanical properties.

The irradiation of polymeric materials with ionizing radiation (gamma rays, X-rays, accelerated electrons, ion beams) leads to the formation of very reactive intermediates, free radicals, ions and excited species. These intermediates can follow several reaction paths that result in disproportion, hydrogen abstraction, arrangements and/or the formation of new bonds. The degree of these transformations depends on the polymer structure and the conditions of treatment before, during and after irradiation [**Chmielewski A.G.,et al., 2005**].

In the radiation induced process, the formation of free radical sites on the polymer is not dependent on temperature but is only dependent on the absorption of the penetrating high-energy radiation by the polymer matrix, therefore, radiation processing is temperature independent or, in other words, we may say it is a zero activation energy process for initiation. As no catalyst or additives are required, the purity of the processed products can be maintained. With radiation processing, the molecular weights of the products can be better regulated. Radiation techniques also have the capability of initiation in solid substrates. The finished products can also be modifyed by the radiation technique [**Bhattacharya A., 2000**].

This study deals mainly with the utilization of recycled and repulped news print microfibers to reinforce EPDM rubber matrix using ionizing gamma radiations, for inducing bonding in EPDM/news print microfibers composites.EPDM has to be cross-linked and form bonding with newsprint microfibers under the effect of gamma radiation to achieve its optimum mechanical performance in terms of tensile strength, Elongation at break point, stress modulus and toughness,X-ray diffraction and FTIR analysis, so we can find that through the following results.

2. Experimental

2.1. Materials

Ethylene propylene diene monomer (EPDM) used in this study was supplied by Jilin chemicals Industry corporation (China). It has the commercial name of Norborene (ENB), propolene content 4.4%, ethylene content 68.5-74.5% and moony viscosity (ML1+4, 121 ^{0}C) of 75.A commercial grade of Stearic acid was used of molecular weight: 284.49, molecular formula: CH_3 (CH_2)16 COOH, Melting point: 54 °C source, PROLABO, France. Zinc oxide (ZnO) awhite to yellow tined powder, 98.0% purity commercial grade, contains lead sulphate 0.25% maximum and calcium residu 0.15% maximum, source,El Nasr Chemical Co. Egypt. Antioxidant (TMQ),1,2dihydro2,2,4trimethyl quinoline, a brown granular beeds, it's Chemical formula:$(C_{12}H_{15}N)_n$, n=2-4, specific gravity:1.05 g/cm^3, Supplied from intrude chemicals (GMbH), Germany.Both Hydrochloric acid and Sodium hydroxide were supplied by El Nasr Company for Intermediate Chemicals, Abu-Rawash Industrial Zone Giza, Egypt.

2.2. Preparation of news print microfiber from Waste news print papers:

The waste newsprint papers were collected from local market or areas and submitted to recycling using chemical pulping and treatment as follow:the shredded waste news print paper fibers were mixed in a kneader with 2M NaOH solution and subjected to continuous stirring for two hours, then the mixure sieved to void excess sodium hydroxide solution and washed with fresh water several times. The fiber after rinse water, treated again using 2M

HCL solution and continuous stirring for another one hour, the batch was washed several times again using fresh water. The residual water in news print pulped fibers (NPF) batch was removed by filtration and then dried at 100 ^{0}C for 24 hr to complete fiber's dryness. The dried fibers were then grinded and sieved from sieve No. 35,(0.5 mm). Mechanical repulping was performed with the same procedure but without using chemiclas to inveistigate the effect of using chemicals in formation of microfiber, it's dimension and it's consituents and the crystalinity of cellulose before and after chemical repulping.

2.3. Preparation of EPDM/news print microfiberscomposites:

Mixing samples was carried out on a laboratory two rolls mill. The roll dimensions are: Outside diameter: 470mm; working distance, 300 mm; speed of slow roll, 24 rpm, and gear ratio is 1.4:1. The EPDM rubber was passed and mixed through the two rolls without banding at a roll opening of about 0.2mm, then the sample was banded with a mill opening of about 1.5mm. a three to four cuts were made every half a minute to ensure a good mixing. After about 20 minutes, the mixing sheets were rolled out of the two rolls mill and left to cool. Sheets of 1mm thickness were covered from each side by sheets of polyester before being pressed in molds of an electric heating press at 160^{o}C for at least 5 minutes and pressure at 60 kg/cm^2. The pressed sheets were taken off the mold and kept between two sheets of polyester tell subjected to gamma irradiation and or testing. The ingredients were mixed according to the mixing ratio listed in table (1).

Table (1): list of the mixing ingradients of the prepared EPdM/news microprint fiber composites.

Ingradiants (gms)	Blank sample	EPDM-Untreated fiber composites
EPDM	100	100
ZnO	5	5
Stearic acid	2	2
TMQ	0.5	0.5
News print microfibers	0	Ranged from5-50

2.4. Gamma irradiation

Gamma irradiation of samples was carried out in the Cobalt-60 source of gamma Chamber-4000A (installed at National Center for Radiation Research and Technology, NCRRT, Cairo, Egypt). It was manufactured by Atomic Research Center, Bombay, India. The composites were subjected to gamma irradiation in air, at room temperature. The irradiation was carried out at a dose rate of ≈ 2.8 kGy /h.

2.5. Characterization

XRD patterns were obtained by a Rigaku RINT X-ray diffractometer (model DMAX-1200) with Ni-filtered Cu Kα radiation at a generator voltage of 40 kV,a wavelength of 1546 nm at room temperature. The XRD diffraction patterns were recorded in the reflection mode in the range of 5° to 90°, with a scanning speed and step size of 2°/min and 0.05°, respectively. Morphology and fiber size were measured using scanning electrin microscopy (SEM) images. SEM micrographs of repulped and treated watse news print fibers was obtained with a JSM-100CX (Shimadzu Company, Japan) at an acceleration voltage of 80 kV. The IR spectra were performed by using FT-IR spectrophotometer (Mattson 100 Unicam, England) over the wavenumbers range 4000-500 cm^{-1}. The thermogravimetric analysis (TGA) was carried out using a TGA-50 instrument from Shimadzu (Japan). The heating rate was carried out at 10^{o}C/min under nitrogen gas atmosphere from room temperature to 500^{o}C.

2.6. Mechanical and physicomechanical properties

Five individual dumbbell-shaped specimens were cut out from the sheets using a steel die of standard width, 4 mm. A benchmark of 1.5 cm was made on the working part of each test specimen. The ultimate tensile strength *(TS)*and elongation at break point*(E_b)* were determined at crosshead speed 50 mm/min on a rubber tensile testing machine, Mecmesin, United Kingdom. Type of multi Test 25-I, provides high capacity force measurements up to 25 kN, with full programming capability. AlsoTensile modulus at 100% Elongation, (M_{100}) and Tensile Toughness (T_t) and crosslinking density were calculated.

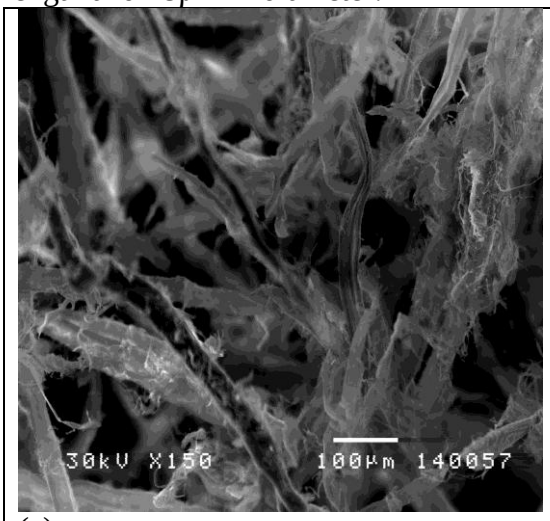
3. Results and disscusion:

3.1: Characterization of news print fibers:

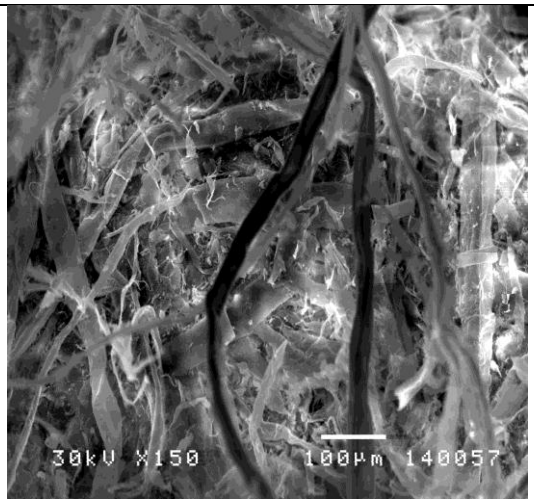
3.1.1. Fiber type and dimensions (Scanning Electron Microscopy):

The fibers used in this study were prepared using chemical repulping of waste news print papers as mentioned in the experimental part. These fibers grinded after drying to pass from 0.5mm sieve. Mechanical repulping for news print fibers was made to compare fiber properties before and after chemical treatment or repulping.

Figure (1) illustrate scanning electron microscopy (SEM) of both mechanical and chemical repulped, grinded news print microfibers enlarged for 150 times to determine its dimensions on a scale of 100 micron.. It was noticed that the news print microfibers in image have some flashes and residues on their surface; this may be due to vigorous stirring kneader. Image dimension analysis using, imagj program, was performed on the previous images, it was found that the average relative dimensions of the news print microfibers as mentioned in table (2). The mechanical repulped fibers have the dimensions ranged between 622µm to 355µm in length and from 40µm to 17µm in width or diameter with an average 486µm in length and 28µm in diameter.



(a)



(b)

Figure (1) scanning electron microscopy of (a) Mechanically repulped news print fibers (MR-NPF) and (b) chemically repulped News print fibers treated (CR-NPF), magnified at 150X.

Table (2): different dimensions of news print fibers (NPF), after mechanical and chemical repulping.

Sample ID		Maximum. µm	Minimum. µm
MR-NPF	Length	622	355
Average		***486***	
	Width	40	17

Average		***28***	
CR-NPF	Length	511	221
Average		***384***	
	Width	33	7
Average		***19***	

It was also noticed that the chemical repulped news print microfibers has less dimensions than that of mechanical repulped one. SEM microscopy reveals that the chemical repulped news print microfiber has the dimensions ranged from 511µm to 221µm in length and from 33µm to 7µm in width or diameter with an average of 384µm in length and 19µm in width or diameter. This results indicated that the chemical repulping make the news print fibers thinner and shorter.

3.2. X-ray Diffraction:

3.2.1.X-ray diffraction parameters

Figures (2a) and (2b) show the x-ray diffraction peaks of the mechanical (MR-NPF) and chemical (CR-NPF) repulped news print fibers. ***For mechanical repulping news_print fibers*** the diffraction peaks appearing at 2θ, 15.34 and 16.58 which assigned for (110) and (110) crystallographic planes. The peaks at 2θ, 22.85 and 34.64 reflections are assigned to the (200) and (400) crystallographic planes. The peaks at 2θ, 29.48, 37.71, 43.86 and 47.74 are assigned for TiO_2 and PbO residue exist in printing ink mixed with the news print fibers [**Matheus Poletto et al., 2013**]. The x-ray diffraction pattern of ***chemical repulped_news print fibers*** characterized by peaks at 2θ 15.49 and 16.74which assigned for the (110) and (110) crystallographic planes, peaks at 2θ 23.15 and 34.63 for both (200) and (400) crystallographic planes. The foure peaks corresponding to the crystalline regions as cellulosic material [**Davidson T.C, et al., 2004and Park Sunkyu et al., 2010**]. Peaks at 2θ, 29.48, 37.71, 43.86 and 47.74 appears previously in mechanical repulped fibers are found to disappear in the x-ray pattern of news print fibers after chemical repulpimg. This may due to some chemical reaction for residual oxides in the printing ink with HCL acid during chemical repulping. The X-ray diffraction peaks for the mechanical and chemical repulping news print fibers are summarized in table (3).

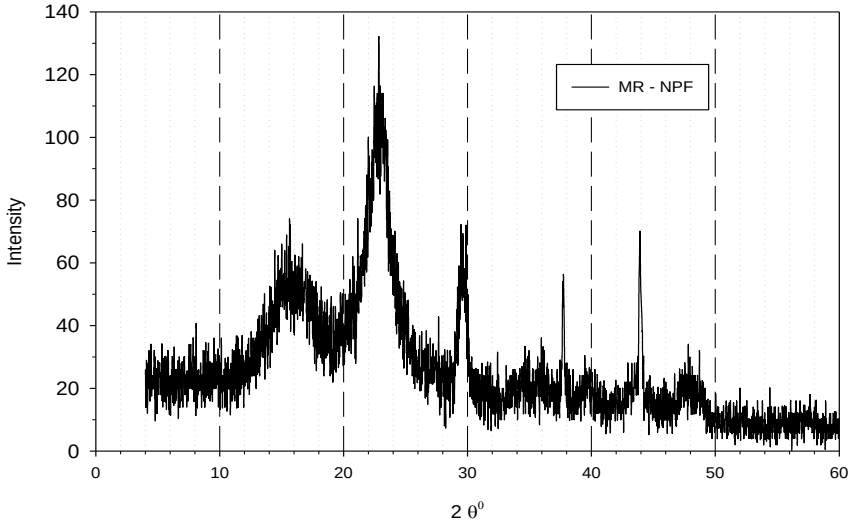


Figure (2a): X-Ray Diffraction pattern of mechanically repulped news print microfibers

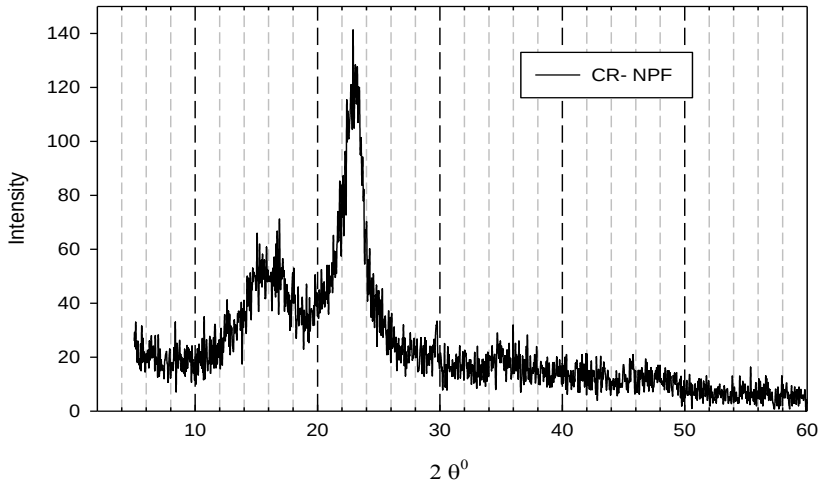


Figure (2b): X-Ray Diffraction pattern of chemically repulped news print microfibers

Crystallinity index (Cr.I.) of news print microfiber samples was calculated using the XRD data using height method which developed by Segal and coworkers and other papers [**Gumuskaya E., 2003and Segal L. et al., 1962**]. The proposed method was for empirical measurements to allow rapid comparison of cellulose samples. Crystallinity index (Cr.I.) was calculated as presented by equation (Eq. 1).

$$\mathbf{Cr.I. = (I_{200} - I_{AM}) / I_{200}} \qquad \mathbf{(1)}$$

Where: I_{200} is the maximum intensity of the (002) lattice diffraction and I_{AM} is the intensity diffraction at about 18-19 2θ degrees. [**Matheus Poletto et al., 2013,Popescu M.C., et al., 2011**]. From the ratio of the height of the 200 peak (I_{200}) and the height of the minimum (I_{AM}) between the 200 and the 110 (at 18.65-19.10 $2\theta^0$) peaks, as shown in Figures 2a and 2b. The height ratio between the intensity of the crystalline peak (I_{200} - I_{AM}) and total intensity (I_{200}) after subtraction of the background signal measured without cellulose [**Bateman L., 1963and Zang Y. H., et al., 1986**], this method is useful for comparing the relative differences between samples.The *d*-spacings were calculated using the Bragg equation (Eq. 2), [**Kim U.J., et al. 2010 andWada M., et al., 2001**].

$$\boldsymbol{n\lambda = 2dsin\theta} \qquad \mathbf{(2)}$$

where: *n* is the order of reflection, λ is the wavelength of the incident X-rays, *d* is the interplanar spacing of the crystal and θ is the angle of incidence. The apparent crystallite size (L), was calculated using the ***Scherrer equation***, (Eq. 3), [**Matheus Poletto et al., 2013, Popescu M.C., et al., 2011**]:

$$\boldsymbol{L = (K\ x\ \lambda)/(H\ x\ cos\theta)} \qquad \mathbf{(3)}$$

Where: *K* is a constant of value 0.94, λ is the X-ray wavelength (0.1542 nm), *H* is the half height width of the diffraction band and θ is the Bragg angle corresponding to the (200) plane.

Table (3): X-ray difraction peaks ($2\theta^o$) of the news print fibers after using mechnical rpuping (MR-NPF) and chemical rpulping (CR-NPF)

Sample	Assigned for (110) plane	Assigned for (110) plane	Assigned for (200) plane	Assignedfor (400) plane
MR -NPF	15.34	16.58	22.85	34.64
CR-NPF	15.49	16.74	23.15	34.63

The x-ray diffraction band position $2\theta^0$ and d-spacing of crystalline and amorphous cellulose regions of both mechanically and chemically repulped news print microfibers are given in table (4). It was noticed that the values of band position and d-spacing, table (4), were approximately similar for both mechanically (MR-NPF) and chemically (CR-NPF) repulped news print fibers. But the d-spacing values for CR-NPF are slightly lower than that of MR-NPF, This may be due to the chemical repulping and hence enhancing crystallinity of cellulose as presented in table (5).

Table (4): band position (2θ) and d-spacing of crystalline and amourphous cellulose regions for mechanically (MR-NPF) and chemically (CR-NPF) repulped news print fibers.

Sample	(110) plane		(110) plane		Amorphous	(200) plane	
	2θ	*d- (nm)*	2θ	*d-(nm)*	2θ	2θ	*d- (nm)*
MR-NPF	15.34	0.618	16.58	0.543	18.93	22.85	0.390
CR-NPF	15.49	0.598	16.74	0.537	18.85	23.15	0.385

Table (5) Crysallinity indeces and crystal size of both mechanically (MR-NPF) and chemically (CR-NPF) repulped news print fibers.

Sample	Amorphous		(200) plane		Crystallite size (nm)	Ceytallinity index (%)	Z-value
	2θ	Intensity	2θ	Intensity			
MR-NPF	18.93	33	22.85	115	4.3	71.5	
CR-NPF	18.85	31	23.15	128	8.3	75.78	

The degree of cellulose crystallinity is one of the most important crystalline structure parameters. The rigidity of cellulose fibers increases and their flexibility decreases with increasing ratios of crystalline to amorphous regions [**Gumuskaya E., et al., 2003**]. The crystallinity index and crystallite size calculated showed that the CR-NPF crystallinity and crystallite size are higher than that of MR-NPF, as presented in Table (5). These differences are confirmed when the values of the crystallite size along the three crystallographic planes are taken into consideration. Crystallinity indices increased with increasing crystallite sizes because the crystallites surface corresponding to amorphous cellulose regions diminished, [**Kim U.J., et al. 2010**].

The crystallinity index (Cr.I.) shows a considerable difference (about 5%) in crystallinity between the two news print fiber samples. However, the increase in the crystallite size for CR-NPF in (110) might be associated with a reduction in the corresponding amorphous region [**Kim U.J., et al. 2010, Newman R.H., 1999**]. If the amorphous domains of cellulose are attacked during the chemical repulping treatment, chain scission and peeling reactions can occur, which reduce the total amount of amorphous regions and therefore increase the CR-NPF crystallite size hence its crystallinity index.

These results confirm that chemical repulped news print microfibers contain more cellulose chains in a highly organized form than the mechanically repulped news print microfibers. This can lead to higher hydrogen bond intensity among neighboring cellulose chains resulting in a more packed cellulose structure besides higher crystallinity.

3.3. FTIR analysis:

The infrared transmittance bands of the mechanical (MR-NPF) and chemically (CR-NPF) repulped news print fibers are represented in figures (3a) and (3b) respectively. When comparing the two spectra, it was found evidence that chemical cellulose was transformed to a more dense and crystalline structure by the effect of chemical rpulping. The differences in spectra, between mechanically and chemically repulped news print fibers were explained in table (6). It was observed that there are two main absorbance region, these regions are found

at low wavenumber from 416 to 1794 cm^{-1} and the other at high wavenumber from 2850 to 3750 cm^{-1}.

The low wavenumber region of these spectra in both figure (3a) and (3b) characterized by stretching vibration at 416-580 $cm^{-1}$of different C-C bonds in some free β D-glucose units [**Zhbankov R.G., et al., 1997**], that may exist accompanied with news print fiber due to agitation during mechanical repulping. These bands were fused in two bands at 450 and 580 cm^{-1} due to chemical repulping. The bands at 624, 687, 704 and 756 cm^{-1} are assigned for out of plane bending vibration of C-OH, out of plane deformation of OH⋯H hydrogen bonding, and rocking vibration of CH_2 respectively in cellulose chains. The bands at 809 and 874 cm^{-1} are assigned for the C-O and C-H deformation vibration in cellulose molecules. These bands were found to disappeared or fused due to chemical repulping this as a result of reordering and recrystallization of cellulose molecules [**Sang Youn Oh., etal., 2005, Anuj Kumar, et al., 2014**]. The band appears at 904 cm^{-1} is assigned for the asymmetric rocking of C-H of the β-glucosidic linkage in amorphous cellulose, hence it's disappearance due to chemical repulping means that amorphous cellulose transform to crystalline one. This explanation would supported by the increase in the intensity (as shown in figures 3a and 3b) of the band at 1054 cm^{-1} which assigned for the C-O-C (β-glucosidic linkage) stretching vibration of cellulose ring in crystalline cellulose phase due to chemical repulping [**Mohamed M.A.,et al., 2105**]. The vibrational band at 1113 cm^{-1} is assigned for O-H asymmetric vibration in cellulose and hemiclellulose which is reduced as result of dissolution of hemiecelluose using chemical repulping. The bands at 1155, 1321 and 1382 cm^{-1} are assigned for C-O-C asymmetric stretching vibration of β-glucosidic linkage and C-H bending asymmetric deformation vibration in cellulose respectively. These bands reduced as a result of reordering and recrystallization of cellulose molecules.

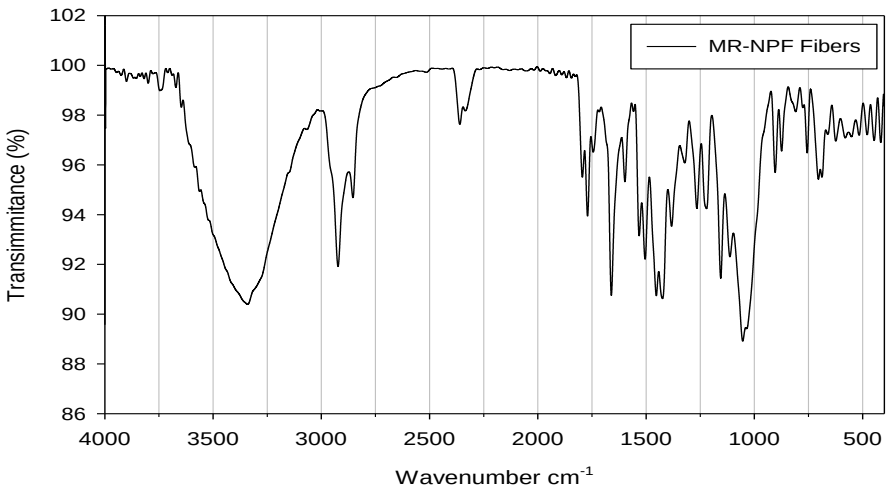


Figure (3a): FTIR analysis of chemicall repulped news print microfibers

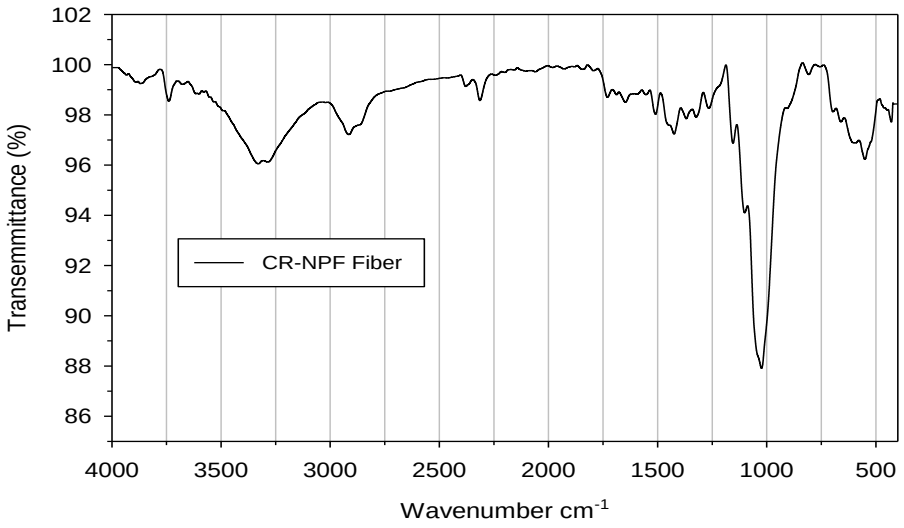


Figure (3b): FTIR analysis of chemically repulped news print microfibers

The bands at 1221 and 1265 cm^{-1} are assigned for CH-O and C-O stretching vibrations in lignin. The bands at 1424 and 1532 cm^{-1} are assigned for C-H deformation vibration and C-H bending vibrations of aromatic compounds in lignin respectively. The bands at 1504 and 1598 cm^{-1} are assigned for C=C in plane symmetrical vibration and unsaturation linkage of aromatic rings in lignin. Also the band at 1452 cm^{-1} and the bands 1744-1794 cm^{-1} are assigned for in plane symmetrical vibration of C=O in lignin and for acetyl ester C=O groups of hemicellulose along with eater linkage of carboxylic groups of ferulic acid in lignin respectively[**Anuj Kumar, et al., 2014- Ziaul Karim Md., et al., 2014**].

Table (6): FTIR spectra of mecnichally (MR-NPF) and chemically (CR-NPF) repulped news print fibers

Wave No. (cm^{-1})	***Mechanical repulped news print fibers, (MR-NPF)***	***Chemically repulped news print fibers, (CR-NPF)***
416-580	Assigned for stretching vibration of different C-C bonds in β-D-glucose unit of cellulose (32)	Fused in tow bands at 450 cm^{-1} and 580 cm^{-1}, other bands disappered due to recrystalization of cellulose.
624	Assigned for C-OH out of plane bending vibration	Disappeared due to chemical repulping
687,704	Both are are assigned for out of plane deformation vibration of OH⋯H hydrogen bonding (27, 28)	Disappeared due to chemical repulping
756	Assigned for rocking vibration of CH_2 in cellulose.	Disappeared or fused with other bands due to chemical repulping
809,874	Both are assigned for C-O and C-H deformation vibration in cellulose chains	Disappeared or fused with other bands due to chemical repulping
904	Assigned for the asymetric rocking of C-H of the β-Glucoside linkage in ***amorphous cellulose*** (29, 30).	Disapeared due to chemical repulping and recrystalization of cellulose (29, 30).
1054	Assigned for C-O-C streching vibration of cellulose ring in	its intensity increased due to chemical repulping and hence

	crysatlline cellulose (29).	increase in cellulose content (29).
1113	O-H assymetric vibration in cellulose and hemicellulose	Reduced due to chemical repulping
1155	Assigned for C-O-C asymetric stretching of β-Glucoside linkage (27, 29).	Reduced due to chemical repulping
1221	Assigned for CHO stretching in lignin (28-31).	Disapeared due to removing of liging residue (28-31).
1265	Assigned for C-O streching vibration in lignin	Reduced and fused due to removing of liging residue
1321, 1382	Both assigned for C-H bending asymetric deformation vibration in cellulose	Reduced due to chemical repulping
1424	Assigned for C-H deformation vibration in lignin	Reduced due to removing of liging residue
1452	Assugned for C=O vibration in lignin in plane (27, 29).	Reduced due to removing of liging residue (27,29).
1504	Assigned for C=C in plane symetrical stretching vibration of aromatic ring in lignin (29).	Reduced due to removing of liging residue (29).
1532	Assigned for aromatic compounds C-H bending stretching vibration in lignin	Reduced due to removing of liging residue
1598	Assigned for C=C unsaturated linkage in aromatic rings in lignin	Disappeared due to removing of liging residue
1660	Assigned for O-H deformation stretching vibration from absorbed water	Reduced due to chemical repulping
1744-1794	Assigned for acetyl ester C=O groups in hemicellulose along with ester linkage of carboxylic groups of ferulic acid of lignin (28, 29).	Reduced and disappeared due to removing of hemicellulose and liging residue (28, 29).
2854	Assigned for =CH asymetric stretching vibration in lignin	Disappeared due to the removal of lignin
2923	Assigned for =CH symetric stretching vibration in cellulose (28, 29)	Its intensity reduced due to chemical repulping
2995-4000	Broad band reach its amximum at 3340 cm^{-1}, assigned for interamolecular O-H stretching vibration including hydrogen bonding in cellulose (27-29).	Its intensity reduced dramatically due to breaking of hydrogen bonding and recrystalization of cellulose (27-29).
3750	O-H streching vibration of terminal molecules in cellulose chains.	It's intensity increased.

All of the previous bands were reduced or disappeared as a result of removing of lignin residue in the news print fibers. Also the band at 1660 cm^{-1} is assigned for O-H deformation stretching vibration of the absorbed water in cellulose.

The high wavenumber region which lay from 2850 to 3750 cm^{-1} is characterized by the vibration band st 2854 cm^{-1} which assigned for =CH asymmetric stretching in lignin, this

band was also reduced or almost disappeared as a result of removing of lignin. The bands at 2923 cm^{-1} assigned for =CH symmetric stretching vibration in cellulose which reduced due to chemical repulping. The broad band 2995-4000 cm^{-1} which reaches its peak at 3340 cm^{-1}, assigned for interamolecular O-H stretching vibration including hydrogen bonding along cellulose chains backbone [**Sang Youn Oh., et al., 2005and Mohamed M. A., et al., 2015**]. Refer to figures 3a and 3b, it was noticed that the intensity of this band was reduced dramatically as a result of breaking of interamolecular hydrogen bonding and reformation and recrystallization of cellulose molecules due to chemical repulping. Finally the band at 3750 cm^{-1} assigned for the free –OH groups along cellulose terminal molecules of the microfibers which repulped mechanically or chemically, but it became more intense in chemically repulped news print microfibers.

It can be concluded that cellulose in chemically repulped news print microfibers became highly crystalline than that of the mechanically repulped one due removing lignin and hemicelulose residue and in the same time due to reordering and recrystallization of cellulose molecules and hence recrystallize it's chains.

3.4. Gamma irradiation cured news print fibers/EPDM composites:

3.4.1..Mechanical properties:

3.4.1.1. Tensile strength (TS):

The effect of gamma irradiation dose and fiber concentration on tensile strength (TS) of the EPDM/news print fiber composites prepared using different fiber concentration namely 5, 10, 20, 30, 40 and 50 phr, was shown in figures (4) and (5). In figure (4) It can be seen that values of TS tend to increase with the increasing absorbed dose up to 40 kGy, then decrease slowly with increasing absorbed dose tell reach 100 kGy. The effects of ionizing radiation cause of crosslinking and degradation reactions in elastomers[**Bateman L., et al., 1963**].

The cross-linking caused increasing mechanical properties up to a level of 40 kGy in this case. Whereas degradation reaction caused a decrease in mechanical properties as shown in figures.The results show that EPDM samples without fibers have the lowest values of tensile strength (TS). Whereas the tensile strength values of the EPDM/fiber composites tend to decrease with increasing fiber concentration gradually. Also the values of TS were found to dramatically decrease after 10 phr fiber content, figure (5). This behavior is may be attributed to cellulosic news print micro fibers act as a reinforcement points through achieving a proper fiber distribution within the EPDM matrix and also in presence of some kind of bonding as a result of applying irradiation. Hence the resistance to the force applied transfer from the well distributed fibers to the EPDM matrix. Hence the tensile strength values increased and reach maximum at fibers content reach 10 phr,after this ratio tensile strength decreased with increasing fiber content as a result of fiber agglomeration due to increasing its content.

Then adhesion decreased and bonding brcame weak between excess fiber content and EPDM polymer matrix. This leads to deteriorating tensile strength values tell reachimg minimum at 50 phr fiber concentration as shown in figure (5).

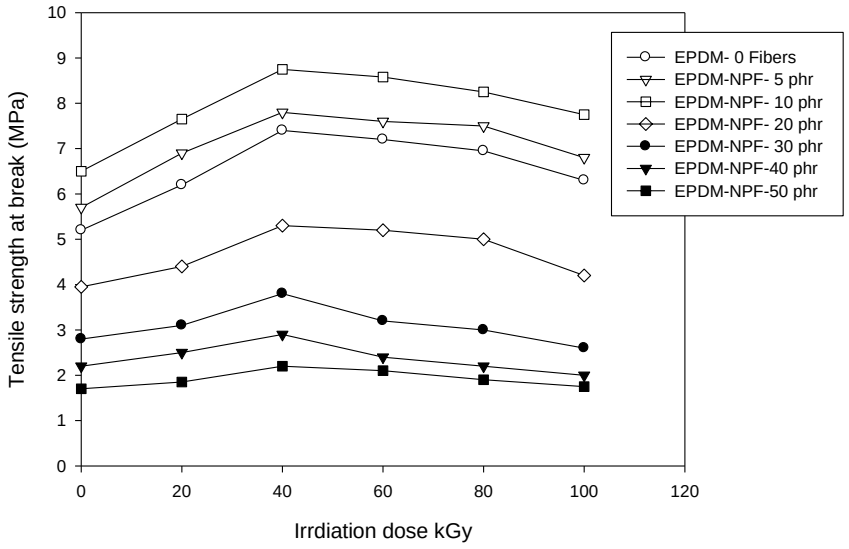


Figure (4): Effect of γ–rays irradiation dose on tensile strength of EPDM-news print fiber composites prepared using different fiber concentration.

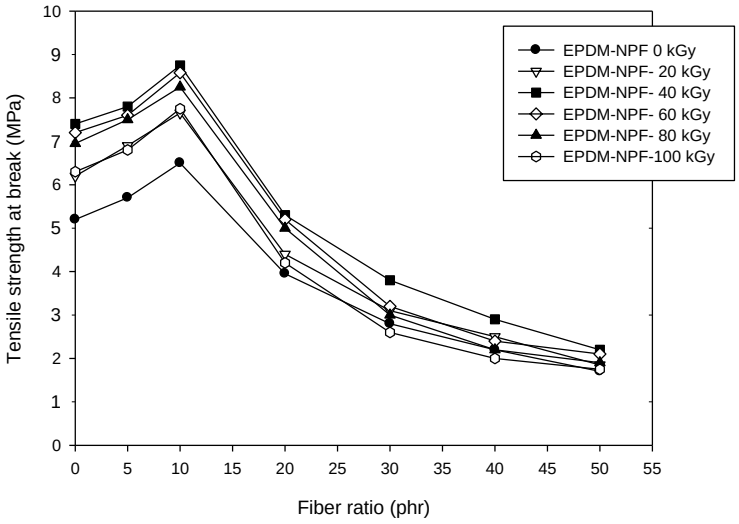


Figure (5): Effect of News print fiber concentration on tensile strength of EPDM-news print fiber composites prepared using different γ–rays irradiation dose

Figure (5) shows that the EPDM/news print fiber composites have the highest tensile strength values at 40 kGy and the lowest for the un-irradiated EPDM/news print fiber composites. It was also noticed that the TS values are approximately equals to 5, 10, 20 phr of fiber for both 20 and 100 kGy irradiation dose, this may be as a result of initiated crosslinking reactions due to the energy absorbed in contrast of initiated degradation reaction after excess energy absorbed for 100 kGy. All other TS values were found to be slightly different than each other except its values at 40 kGy, which were found to exceed all TS values at different irradiation dose and fiber ratio.

The results concluded that tensile strength (TS) values of EPDM/ news print fiber composite reach maximum using 10 phr fiber concentration and 40 kGy gamma irradiation dose. Increasing fiber concentration more than 10 phr leading to decreasing tensile strength (TS). Increasing gamma irradiation dose more than 60 kGy leads to decrease tensile

strengthof the composite. Adding news print fibers to EPDM rubber leads to enhance its tensile strength.

3.4.1.2. Elongation at break point %, (E_b):

Figures (6) and (7) illustrate the variation of $E_b$with gamma irradiation dose and fiber concentration of the EPDM/news print fiber composites prepared using different fiber concentration namely 5, 10, 20, 30, 40 and 50 phr. It was noted that the E_b values were slightly decrease with increasing irradiation dose but sharply decrease with increasing fiber concentration.

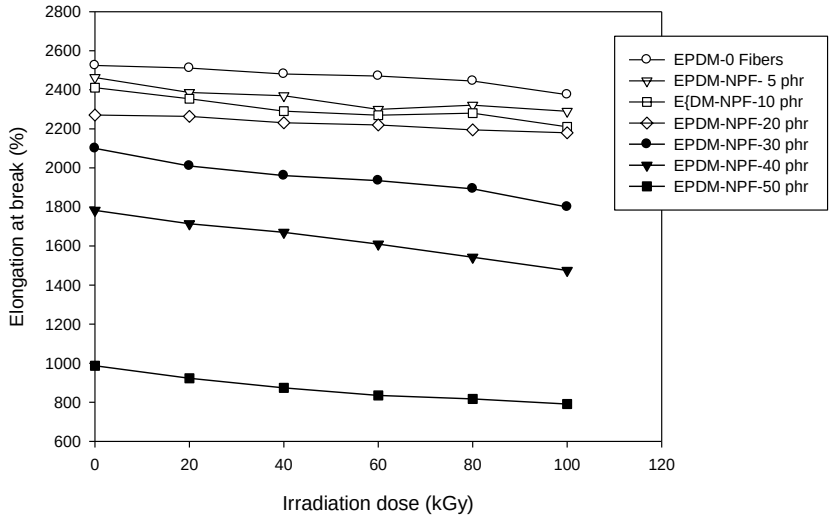


Figure (6): Effect of γ–rays irradiation dose on Elongation (%) EPDM-news print fiber composites prepared using different fiber concentration.

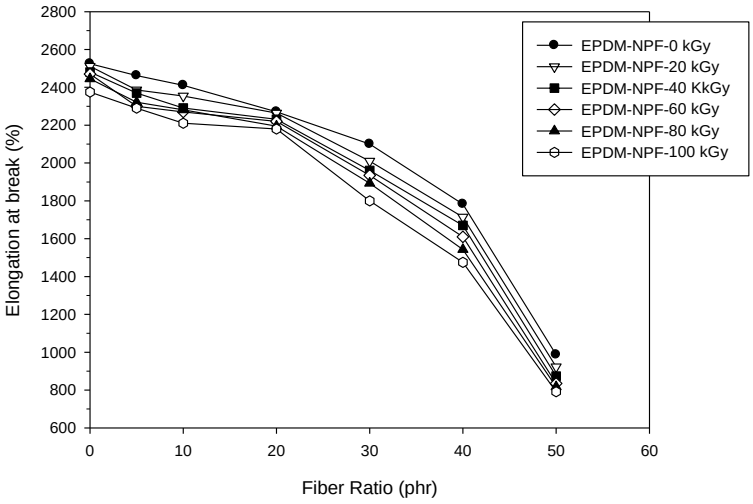


Figure (7): Effect of News print fiber concentration on Elongation (%) of EPDM-news print composites prepared using different γ–rays irradiation dose.

These results indicated that the decrease in the $E_b$values with increasing irradiation dose is due to effective increase in crosslinking density at higher doses. Increasing crosslinking density means increasing bonding between polymer chains which renders them incapability of stretching upon deformation, hence decreasing elongation E_b and increasing tensile strength. Whereas, the sharp decrease in E_b values with increasing fiber concentration, is due to restrictions on movement of EPDM molecules, hence lower stretching upon

deformation with applied force, as a result of increasing fiber ratio[**Ali Magdy. A., et al., 2011**]. So, in general the E_b values of EPDM/news print fiber composite were found to decrease under two causes, one, is increasing in irradiation dose and the other is increasing fiber concentration but was not affected in the same way.

From figure (7), the results have shown that the rate of decrease of E_b values was found to be slow from 0 to 20 phr fiber concentration. Whereas, this rate was found to sharply decrease from after 20 phr fiber concentration. Also the results show that the highest values of E_b were belong to EPDM samples without news print fibers and the lowest ones belong to that samples contain 50 phr news print fiber content. This may attributed to the reasons mentioned above. The results show also that the E_b values of the EPDM/news print fiber composites slightly decrease with increasing fiber content from 5 to 20 phr then it was found to sharply decrease after 20 phr fiber content whereas it was not greatly affected by increasing irradiation dose.

The results concluded that Elongation at break point (E_b)values of EPDM/news print fiber composite decreasing with increasing fiber content especially after 10 phr content of fiber. EPDM samples without fibers have the highest elongation values and decreasing with increasing fiber content and gamma irradiation dose more than 60 kGy as a result of increasing degradation.

3.4.1.3. Tensile modulu at 100% Elongation, (M_{100}):

The correlation between tensile strength at a given elongation, M_{100} and the degree of crosslinking is given by the following equation.

$$F/A_o = \rho R T M_C^{-1} (\lambda - \lambda^{-2}) \quad (4)$$

Where A_o is the cross-sectional area, F is force required to produce elongation λ, ρ is the density of rubber, R is gas constant, T is absolute temperature and M_c is molecular weight between two crosslinks. When ρ, T and λ are kept constant, then F/A_o depend on M_C^{-1}, i.e., the stress at a definite elongation is inversely proportional to M_C, which is related directly to the crosslink density (number of network chains per unit volume) by inverse proportion[**Zang Y. H., et al., 1986**].

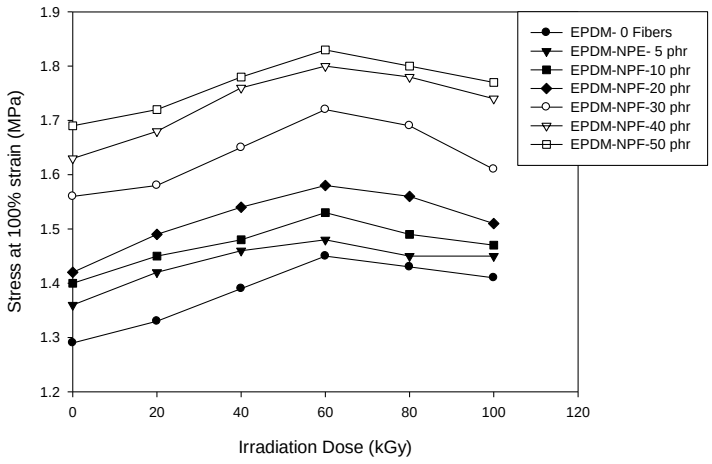


Figure (8): Effect of γ–rays irradiation dose on Stress at 100% strain of EPDM-news print fiber composites prepared using different fiber concentration.

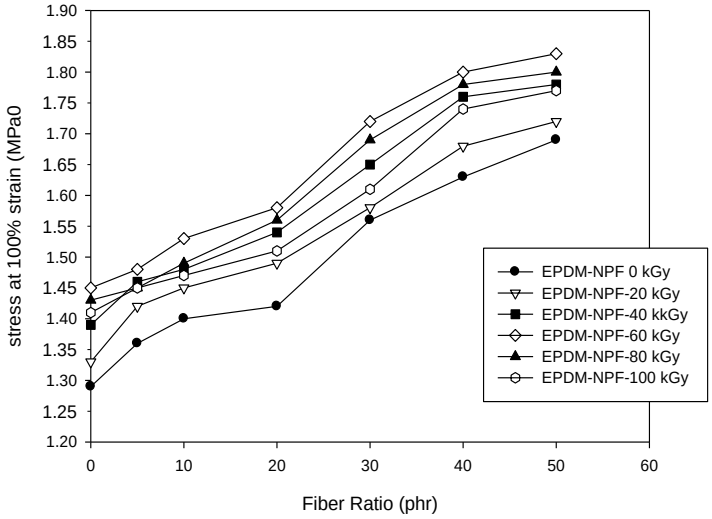


Figure (9): Effect of News print fiber concentration on Stress at 100% strain of EPDM-news print fiber composites prepared using different γ–rays irradiation dose

Figures (8) and (9) illustrate the variation of the tensile modulus at 100% strain, M_{100},with gamma irradiation dose and fiber concentration of the EPDM/news print fiber composites prepared using different fiber concentration namely 5, 10, 20, 30, 40 and 50 phr. It was noted that the M_{100} values increase gradually with increasing irradiation dose up to 60 kGy then slightly decrease with increasing irradiation dose after 60 kGy.

From figure (8), results shown that the EPDM/newsprint fiber composite which contains 50 phr, fiber has the highest values of tensile modulus and decreasing continuously with increasing fiber content. It was also noticed that EPDM samples without fibers have M_{100} values higher than that of the samples contains 5 and 10 phr fiber content, this may be due to that the force applied was not distributed uniformly along the chains of EPDM polymer.

Figure (9) show that the M_{100} values of the EPDM/newsprint fiber composites increase with increasing fiber concentration for allsamples. This result may be arise from that the fiber work asa somehow linking bridges between polymer chains, then lead to increase the resistance to force applied tell stress reach the value of 100% strain. This resistance applied to previous extent or tensile modulus, M_{100}, as denoted by the previous equation, is inverselyproportional to M_C, which is related directly to the crosslink density. This means that crosslinking density of the EPDM/newsprint fiber composites will increase with increasing its tensile modulus values M_{100}.

The results concluded that tensile modulus at 100% Elongation, (M_{100}) values of EPDM/ news print fiber composite increase with increasing fiber content. Increasing gamma irradiation dose more than 60 kGy leads to decreasing tensile modulus of the composite. Adding news print fibers to EPDM rubber leads to enhance itstensile modulus at 100% Elongation, (M_{100}).

3.4.1.4. Toughness (T_t):

Tensile toughness, (joule per cubic meter, $J.m^{-3}$), is the ability to absorb mechanical energy up to the point of failure. Toughness is related to the area under the stress-strain curve. In order to be tough, a material must be both strong and ductile. For example, brittle materials (like ceramics) that are strong but with limited ductility are not tough; conversely, very ductile materials with low strengths are also not tough. To be tough, a material should withstand both high stresses and high strains. Generally speaking, strength indicates how

much force a material can tolerate, while toughness indicates how much energy the material can absorb before rupturing.

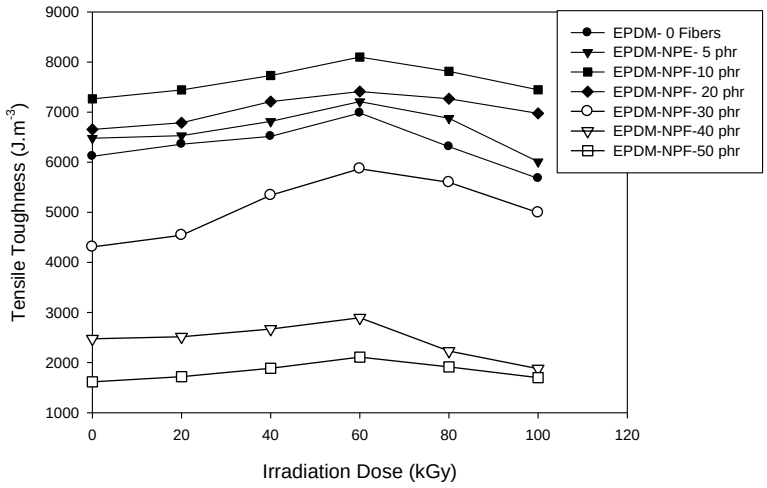


Figure (10): Effect of γ–rays irradiation dose on Tensile toughness of EPDM-news print fiber composites prepared using different fiber concentration.

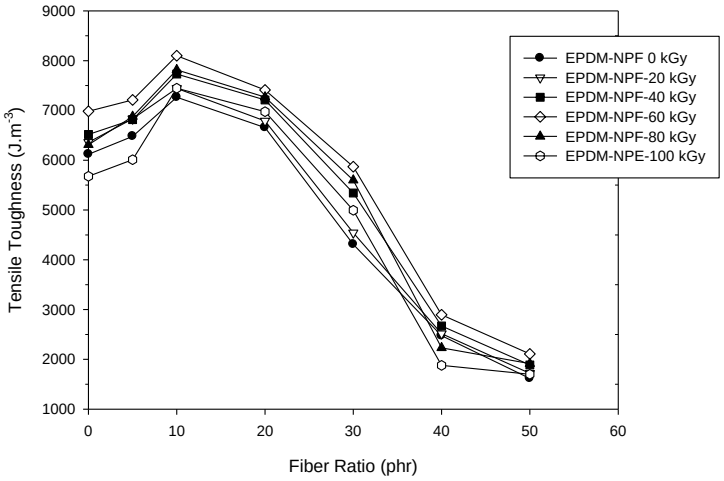


Figure (11): Effect of News print fiber concentration on Tensile toughness of EPDM-news print fiber composites prepared using different γ–rays irradiation dose

Figures (10) and (11) illustrate the effect of gamma irradiation dose and fiber concentration on the tensile toughness, T_t,of theEPDM/news print fiber composites prepared using different fiber concentration namely 5, 10, 20, 30, 40 and 50 phr. the results indicated that that the T_t values increase gradually with increasing irradiation dose up to 60 kGy then slightly decrease with continuous increasing in irradiation dose after 60 kGy. This behavior may be attributed to the occurrence of crosslinking and degradation reactions due to the energy absorbed during gamma rays irradiation. At lowers irradiation doses, from 20 to 60 kgy, Crosslinking reaction leads to increase bonding and in turn results in increasing toughness values. Whereas degradation reactions occur at higher doses, i.e. after 60 kGy, lead to decrease of toughness values.The degradation leads to bond rupture which results in cracks leading to specimen failure at low elongation and stress, [**Ramsteiner F., et al., 2002**].

Also the results indicated that toughness values increase with increasing fiber content up to 10 phr then decline sharpely with increasing fiber content from 20 to 50 phr. this

behavior may attributed to the bonding between polar nature fibers and nonpolar nature of EPDM molecules increase with increasing fiber content to up to10 phr, then tends to decrease with the respect of increasing of polar component of the composites which is the news print fibers, so toughness tends to decrease.

The results concluded that toughness values of EPDM/ news print fiber composite reach maximum using 10 phr fiber concentration and 60 kGy irradiation dose. Increasing fiber concentration more than 10 phr leads to decreasing toughness. Increase gamma irradiation dose more than 60 kgy leads to decreasing toughness of the composite. Adding news print fibers to EPDM rubber leads to enhance its toughness hence its ductility.

3.4.2. Crosslinking density (CD)or υ:

Cross-linking in soft or flexible materials (rubber like) gives a considerable increase in elastic modulus, a marked increase in hardness, and usually a reduction in the ultimate elongation and permanent set. The nature of cross-links plays a big role in determining the physical properties [**Lee S. and Pawlowski H., 1994**]. In other words, crosslink density is an extremely important factor in determining physical properties of vulcanized elastomers.

Elastomers are generally cross-linked in a random manner, therefore, it is difficult to identify the principal effects of modification through mixing of certain components on the mechanical properties. The classical kinetic theory of rubber elasticity originally was developed by Wall, Flory and James and Guth, [**Treloar L. R. G., 1975**]. They attributed the high elasticity of a cross-linked rubber to the change of the conformational entropy of long flexible molecular chains. The theory predicts the following relation in simple extension

$$\sigma = A_{\Phi}\, \upsilon_e\, KT\, (\lambda^2 - \lambda^{-1}) \quad \text{..... (5)}$$

Where σ is the true stress, the force per unit area measured in the strained state, υe is the number of effective plastic chains per unit volume, K is Boltzman`s constant, T the absolute temperature, and λ is the extension ratio; A_{Φ} is a pre-factor depending on the considered model. The elasticity of natural and SBR rubbers in simple extension at constant strain rate. They plotted the true stress as a function of $(\lambda^2 - \lambda^{-1})$ as suggested by the molecular theory. They obtained a series of straight lines which do not pass through the origin[**Zang Y. H., et al., 1986**].

On the other hand, rubber elasticity theory predicts that the relation between the tensile strength and the elongation ratio (24), λ, is;

$$\sigma = \sigma\, 0\, (\lambda) + E\, (\lambda\, 2 - 1/\lambda) \quad \text{..... (6)}$$

Where, σ is the stress, E is the modulus of elasticity and λ is the extension ratio. Figure (5a & b) illustrate the relation between $(\lambda^2 - \lambda^{-1})$ and stress (σ) for NR/SBR and NR/NBR blends. From these figures, it has been calculated the slope of these lines, and then tried to calculate the average molecular weight Mc between crosslinks from the value G according to the well-known, following relation [**Gumuskaya E., et al. 2003**]:

$$G = 3E = A_{\Phi}\rho RT/Mc \quad \text{..... (7)}$$

Where, G is the shear modulus, ρ is the density of the rubber and R the gas constant, the value of Mc, the molecular weight between two crosslink can be calculated and hence the crosslink density υ (or CD) can be calculated from the equation

Crosslink density (υ) = 1/2Mc …… (8)

Hence crosslink density inversely proportion with double the molecular weight between two crosslink. So cross link density found to be directly proportional with the true tensile modulus according to equation (4) and (5).

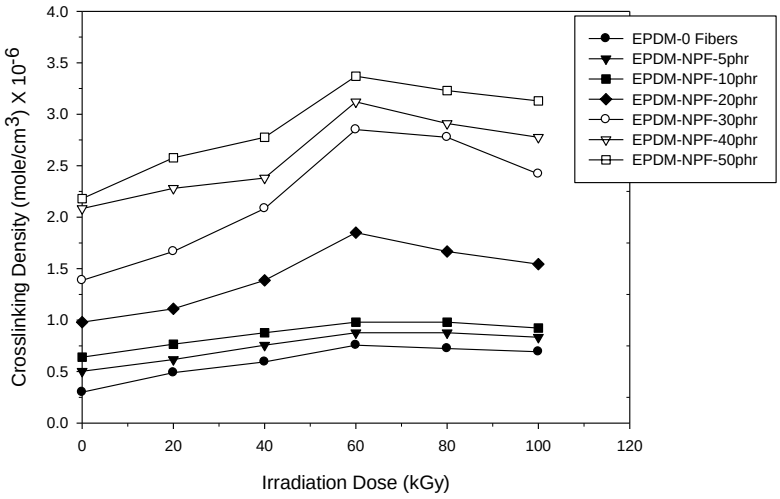


Figure (12): Effect of γ–rays irradiation dose on Crosslinking density of EPDM-news print fiber composites prepared using different fiber concentration.

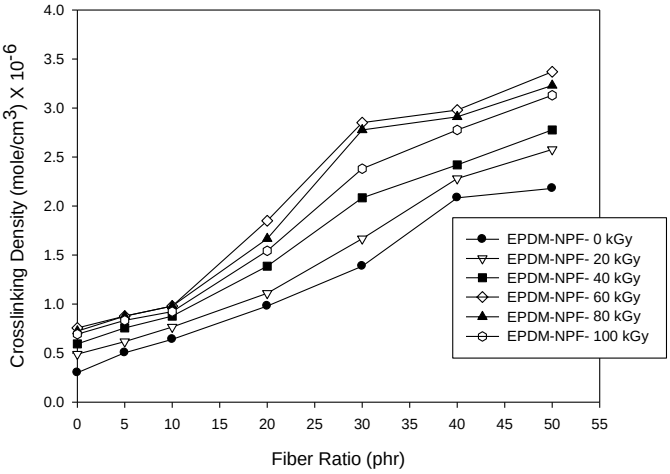


Figure (13): Effect of News print fiber concentration on Crosslinking density of EPDM-news print fiber composites prepared using different γ–rays irradiation dose

Figures (12) and (13) illustrated the effect of gamma irradiation dose and fiber concentration on Crosslinking density (CD)of theEPDM/news print fiber composites prepared using different fiber concentration namely 5, 10, 20, 30, 40 and 50 phr.The results indicated that crosslinking density of the EPDM-news print fiber composites increase with increasing gamma irradiation dose up to 60 kGy then decrease tell reach 100 kGy. Also crosslinking density of the composite increase rapidly with increasing fiber content as shown in figure (13).

The first behavior may be attributed to crosslinking density increase with the increase of the accumulated ionizing irradiation dose up to 60kGy. Of course this will lead to increase bonding through continuous crosslinking reaction between polymer chains and also micro cellulosic fibers. After this dose, 60 kGy, degradation reaction will be the predominant reaction over crosslinking. Hence crosslink density values drop after 60 kGy or become constant in EPDM without fiber, 5 phr and 10 phr EPDM-fiber composite samples, figure (12).This behavior is may be attributed tocellulose classified as a degradable polymer and it is susceptible to degraded after 40 kGy, [Cleland, M. R., et al. 2003].

The second behavior, increasing fiber concentration leads to increasing in crosslink density, may be as result of bond formation between microcellulosic fiber and EPDM polymer chains which in turn crosslink using ionizing radiation. So crosslink density affect by double action in EPDM-news print composite, first bonding occurs in polymer chains by the cause of ionizing radiation and second bonding between microcellulosic fibers and polymer chains which crosslink in the same time using ionizing radiation.

From figure (13), the results shown also that increasing fiber concentration leads to increase crosslink density values without using irradiation dose but have the lowest values, this may be as a result of the formation of some kinds of bonds of physical bonding between microcellulosic fibers and EPDM polymer chains, this kind of bonding increase with increasing fiber concentration. Of course, crosslinking density increase when using ionizing radiation due to enhancing breakage and reformation of bonds between polymer chains and fibers, leading to enhance chemical bonding then crosslinking.

The results concluded that crosslink density increase with increasing irradiation dose up to 60 kGy and continuous increase with increasing fiber concentration up to 50 phr. increasing crosslink density is not beneficial every time, increase crosslink density means increasing bonding then modulus increase and the material become stiffer and less ductile. Increasing modulus leads to decreasing elasticity and increasing tensile strength and low elongation then the material become hard and able to be brittle.

Conclusion:

The results concluded that tensile strength (TS) values of EPDM/ news print microfibers composites reach maximum using 10 phr fiber concentration and 40 kGy gamma rays irradiation dose. Increasing fiber concentration more than 10 phr leading to decreasing tensile strength (TS). Increase gamma irradiation dose more than 60 kGy leads to decreasing tensile strengthof the composite. Adding news print microfibers to EPDM rubber leads to enhance its tensile strength.

Elongation at break point (E_b)values of EPDM/news print microfiber composite was found to decrease with increasing fiber content especially after 10 phr content of fiber. EPDM samples without microfibers has the highest elongation values and decreasing with increasing fiber content and gamma irradiation dose more than 60 kGy as a result of increasing degradation.

Also tensile modulus at 100% Elongation, (M_{100}) values of EPDM/ news print fiber composite increase with increasing fiber content. Increasing gamma irradiation dose more than 60 kGy leads to decreasing tensile modulus of the composite. Adding news print fibers to EPDM rubber leads to enhance itstensile modulus at 100% Elongation, (M_{100}).

The results concluded that toughness values of EPDM/ news print fiber composite reachs maximum using 10 phr fiber concentration and 60 kGy irradiation dose. Increasing fiber concentration more than 10 phr leading to decreasing toughness. Increasing gamma irradiation dose more than 60 kGy leads to decreasing toughness of the composite. Adding news print fibers to EPDM rubber leads to enhance its toughness hence its ductility.

Crosslink density increase with increasing irradiation dose up to 60 kGy and continuous increase with increasing fiber concentration up to 50 phr. increasing crosslink density is not beneficial every time, increase crosslink density means increasing bonding then modulus increase and the material become stiffer and less ductile. Increasing modulus leads to decreasing elasticity and increasing tensile strength and low elongation then the material become hard and able to be brittle.

The x-ray diffraction and FTIR showed that cellulose was recrystalized upon using chemically repulping process and transform to highlly crystalline cellulose chains and that gives the news print fiber and it's cellulosic fibrils higher axial stiffness.

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"تأثير التشعيع الجامى على السلوك الميكانيكى لمتراكبات مطاط الايثلين بروبلين دايين مونمر مع ألياف ورق الصحف المعاد تدويره".

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2-قسم الكيمياء – كلية العلوم (فرع البنات) – جامعة الأزهر.

الملخص العربي

تم تجميع مخلفات ورق الصحف من السوق المحلى وتم معاملاتها كيميائيا باستخدام 2 مولر هيدروكسيد الصوديوم تلاه معالجتها لنفس الفترة الزمنية باستخدام 2 مولر حمض الهيروكلوريك. عقب تجفيف الألياف وطحنا ونخلها يتم خلط الألياف الناتجة مع مطاط الايثلين بروبلين دايين مونمر بنسب مختلفة من 5 الى 50 جزء لكل مئة جزء من المطاط. تم تعريض المتراكبات لجرعات اشعاعيه مختلفة من 20 الى 100 كيلو جراي باستخدام التشعيع الجامى. تم توصيف ألياف ورق الصحف المعاد تدويرها باستخدام الميكروسكوب الإلكتروني الماسح وحيود الأشعة السينية والأشعة تحت الحمراء والتحليل الحراري الو زنى. تم قياس قوة الشد الميكانيكي والإستطاله ومعامل الإجهاد عند استطالة 100 وقوة الصلابة وكثافة الترابط العرضي للمتراكبات المحضرة تحت تاثير تغير نسب الألياف وجرعة التشعيع الجامى. ودلت النتائج على زيادة قوة الشد الميكانيكي بزيادة نسبة الألياف حتى 10 جزء لكل مئة جزء مطاط وزيادة الجرعة الإشعاعية حتى 40 كيلو جراي، بينما تتناقص الإستطاله بزيادة نسبة الألياف وزيادة الجرعه الإشعاعيه. ووجد أن معامل الإجهاد عند استطالة 100 وكثافة الترابط العرضىتتزايد ايضا مع زيادة نسب الألياف حتى 50جزء لكل مئة جزء مطاط وزيادة الجرعة أشعاعيه حتى 60 كيلوجراى. ووجد أن قوة الصلابه لهذه المتراكبات تصل أقصى قيمه لها باستخدام 10 جزء لكل مئة جزء مطاط كنسبة ألياف وباستخدام 60 كيلوجراى كجرعة أشعاعيه.