Study The Properties and Weight Loss Degradation of The Blend LDPE/Cellulose in Soil Environment

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Abstract

Wider applications of polyethylene (PE) in packaging and agriculture have raised serious issue of waste disposal and pollution. Therefore, it is necessary to raise its biodegradability by additives.In this study, we will add cellulose to low density polyethylene to prepare polymer blend have ability to degradation in soil environment. The samples were prepared by using twin screw extruder. LDPE and CELL have been mixing with different weight proportions, and studied their properties in order to determine its compliance with the required specifications to be able to be used biodegradable polymers. To improve the viability of decomposition PEG has been added to the resulting blend. Several tests were applied to identify those properties such as tensile, hardness, density and creep test. FTIR, digital microscope and SEM test acheved in order to determine the miscibility and blend morphology befor and after degradation. The results show that, the blend weight loss increase with increasing CELL percent.

Keywords:LDPE, CELL, PEG, biodegradable, blend, FTIR, SEM, soil burial.

الخلاصة

تطبيقات واسعة للبولي اثيلين جعلت قضية التخلص من النفايات والملوثات ترتفع. وبالتالي، فمن الضروري زيادة التحلل البيولوجي للبولي اثيلين من خلال الإضافات و في هذه الدراسة سوف نضيف السليلوز إلى البولي إثيلين المنخفض الكثافة لتحضير خليط بوليمر يمتلك القابلية على التحلل في التربة العينات تحضر بواسطة باثق ثنائي اللولب حيث يتم خلط نسب مختلفة من البولي اثيلين والسليلوز وتدرس خواصهم لتحديد مدى تطابقها مع المواصفات المطلوبة لتكون قادرة على ان تسنخدم كبوليمرات قابلة للتحلل لتحسين قابلية التحلل يتم اضافة بولي اثيلين كلايكول الى الخليط الناتج. عدة اختبارات تطبق لتحديد هذه الخصائص مثل اختبار الشد والصلادة والزحف التحليل بالاشعة تحت الحمراء والمجهر الرقمي و الفحص المجهري الالكتروني تطبق لتحديد الامتزاجية والهيئة للخليط قبل وبعد التحلل و وتظهر النتائج أن فقدان الوزن للخليط تزداد مع زيادة نسبة السليلوز.

الكلمات المفتاحية: بولي اثبلين منخفض الكثافة ، السليلوز ، بولي اثلين كلايكول ، تحلل ، خليط ، الاشعة تحت الحمراء، الفحص المجهري الالكتروني، اختبار الدفن.

Introduction 1.

The structure of Polymersconsisting of a great number of repeating units and high molecular mass compounds. Now these days, polymer materials are very important in our life daily that entity of human life depends on these polymers. Polymer materials have wide range of properties that can be used in different applications. There has been increasing public concern over the harmful effects of petrolium-based polymer packaging materials specially polyolefins in the environment after the usage. These pectrolium – based polymers create significant amount of waste after the usage and that generates the critical environmental issues. Recycling, recovery and disposal of plastic waste are some of the options available. These methods have certain disadvantages and not hundred percent practical. An important alternative to minimize the polymer waste is the introduction of biodegradable polymers, which can be degraded through the action of naturally occurring microorganisms [Samarasekara ,2013].

There is abundant importance in exchange some or all of the synthetic plastics by natural or biodegradable materials in many used. because a lot of plastics uses in the food industry, even a slight decrease in the amount of materials used for each package would leads to a important polymers droop, and can improve problems of solid waste clearly that the utilize of biodegradable polymers for packaging show an alternatives and partial solution to the problem of accumulation of solids waste consisting of synthetics inert polymers [Behjat ,2009].

Nowadays, It has been accepted blending biodegradable polymers with ineffective polymers as a possibles application in the waste disposal of plastics. In principal, and the way of thinking behind this method is that if the biodegradable sections is present in enough quantity and if microorganisms in the waste disposal environment degrade it, plastics or films containing the residual inert component should loss its integrity, fall to pieces and fades away.[Behjat ,2009].

2. Materials and Sample Prepration

Low density polyethylene (LDPE) was obtained from Amir Kabir Petrochemical Company as granular materials, Iran . Cellulose (CELL) was obtained from Central Drug House (P) Ltd,.Polyethylene glycol (PEG) was obtained from Sinopharm Chemical Reagent Company, Ltd, Chain.

LDPE/CELL blends contain varying ratio according to table, then PEG added for all sample above also show in same table. The mixing process was carried out in twin screw extruder model (SLJ30A) The mixed materials were fed from a hopper on the screw. It's then conveyed along the barrel where it is heated by conduction from the barrel heaters and shear due to its movement along the screw flight. The depth of the twin screw channel is reduced along the length of the screw so as to compact the material. At the end of the extrusion, the melt passes through a die in the form of sheet. The extruder operation conditions were 35rpm in the begning increased to 50 rpm of screw speed. and the temperatures used for zones 1 and 2 were (135-150)°C respectively.



Fig. 1 shows the twin screw extruder.

Table 1.percent ratio of sample

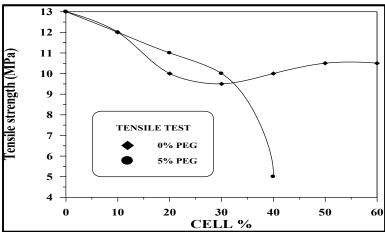
LDPE%	CELL%	PEG%
100	0	0
90	10	0
80	20	0
70	30	0
60	40	0
50	50	0
40	60	0
85	10	5
75	20	5
65	30	5
55	40	5

3. Result and Discussion

3.1 Tensile Tests Results

Figure 2 and 3 show the effects of (CELL) percent on the value of tensile strength and elongation for low density polyethylene. The machine used for the testing of tensile properties is micro computer controlled electronic universal testing machine model (WDW-5E). The results show that the tensile strength and elongation decreases with (CELL) percent increasing, decreasing in elongation and strength of blend due to inadequate wetting of the fibre with the matrix ,uneven aligning of the cellulose fibres and also poor adhesion between the filler and matrix. The poor adhesion between LDPE and CELL lead to form numerous voids at the fibre matrix interface, and the stress transfer to the fibres, which are the load bearing entities, becomes inefficient leading to lower strength and elongation values. These results are agreement with those obtained by [Behjat, 2009]. Precences of (PEG) led to improve values at certain percentage due to closed voids formed of collect cellulose fiber ends as well as the viability wetting fiber by (PEG), It makes PEG works as the surface between the fiber and matrix increase the bonding strength; therefore observe a slight increase in tensile strength and elongation.

At high percent of cellulose, observe a slight increasing in tensile strength while remaining less than pure LDPE due to the penetration of cellulose fiber between the chains which reduces the free movement of chains, which are reflected on the tensile strength.



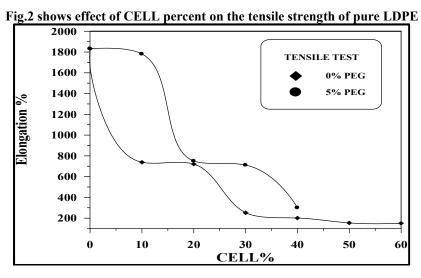


Fig.3 shows effect of CELL percent on the elongation of pure LDPE

The modulus increase with increasing cellulose percent this due to the high modulus of cellulose than LDPE, this result show in figure 4.

Increasing modulus of elasticity of blend compared with matrix polymer due to the limitation of macromolecules on mobility and deformability imposed by the presence of cellulose, since the Young's modulus is a measure of the material stiffness, with increasing cellulose content the materials become stiffer. this result agreement with[Dmitri et.al.,2011]

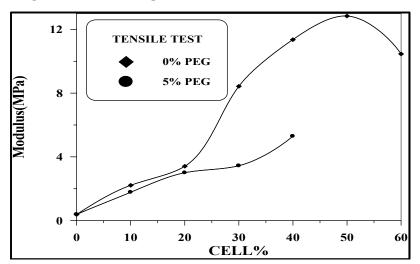


Fig.4 shows effect of CELL percent on the modulus of pure LDP

3.2 Creep Test Result

The machine used for the creep test is WP 600 Kriechtester creep testing machine device. Figs.5 and 6 show (strain-time) curves of LDPE/ CELL and LDPE/PEG /CELL blends respectively. These figures indicate that the creep rates decrease rapidly with the time for all samples, it can also be observed from these figures that the creep rates depend on the (CELL) content. In these curves the creep stages (instantaneous deformation, primary and secondary creeps) can be clearly observed. There is no evidence of tertiary creep, that is, creep rapture, which would require longer time and larger stress. Initially dislocations are generated continually in the primary creep stage. With time, more and more dislocations develop, producing

increasing interference with each other's movement, thus causing creep rate to decrease.

This dynamic equilibrium sets in secondary creep and the material creeps at a constant rate. at low precentage cellulose loading, the microfibrils are sparsely distributed, attraction of the OH groups is minimal. Because of the incompatibility of cellulose and the nonpolar LDPE, the regions occupied by cellulose behave like regions with defects, hence potential sites for crack initiation and propagation.

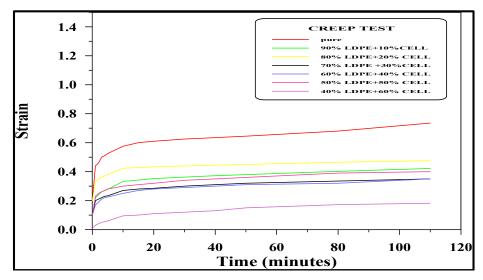


Fig.5 shows (strain-time) curve of pure LDPE and LDPE with CELL in different precent.

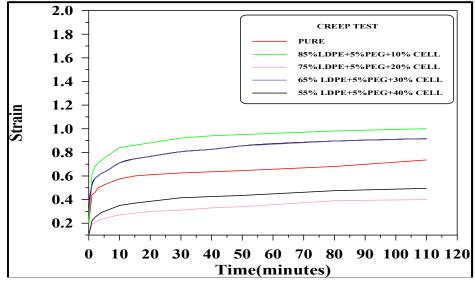


Fig.6 shows (strain-time) curve of pure LDPE and LDPE+5% PEG with CELL in different precent.

3.3 FTIR Analysis

Through this analysis of LDPE/CELL and LDPE/PEG/CELL blend according to bands values, recorded by Fourier transform spectroscopy (FTIR) for the pure polymers and blend prepared by method of extrusion with different composition of blends are summarized in Table 2, which were derived from Figure 7.FTIR test for low density polyethylene shows many bands such as the bands at 2921 and 2849 cm⁻¹

for(C-H stretching), the band at 1465cm⁻¹ for(CH₂ bending), The band at 720 cm⁻¹ for (CH₂ rocking).

FTIR of the blends from (LDPE/CELL) and (LDPE/PEG/CELL) , it was observed shifting spectra to lower wavenumbers ,higher wavenumbers respectively, as show in figure 7.

Results from FTIR test show no any reaction between them ,this due to no bonds appear,while the results show some bonds shifting this due to physical interaction.

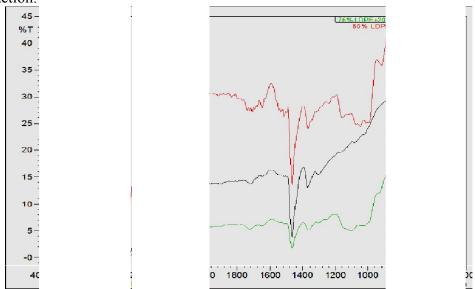


Fig. 7 Shows FTIR for LDPE and LDPE/CELL and LDPE/PEG/CELL Table 2: The absorption bands of the IR spectrum characteristic of LDPE and blends

Type of bond	Stander LDPE [Mark Jordi, J.V. Gulmine]	Exp. LDPE	LDPE+CELL	LDPE+PEG +CELL
CH ₂ stretching	2914 2846	2921 2849	2920 2852	2922 2848
CH ₂ bending	1474	1465	1463	1467
CH ₂ rocking	720-724	720	721	719

3.4 Density Test

figure 8 shows the effect of addition of CELL to LDPE, this figure indicates that the density increase with addition of CELL that is related to the CELL fill the void which has higher density values.

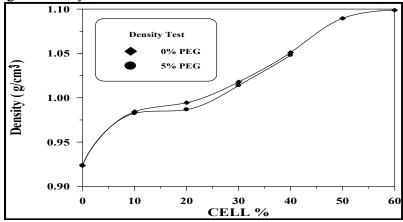
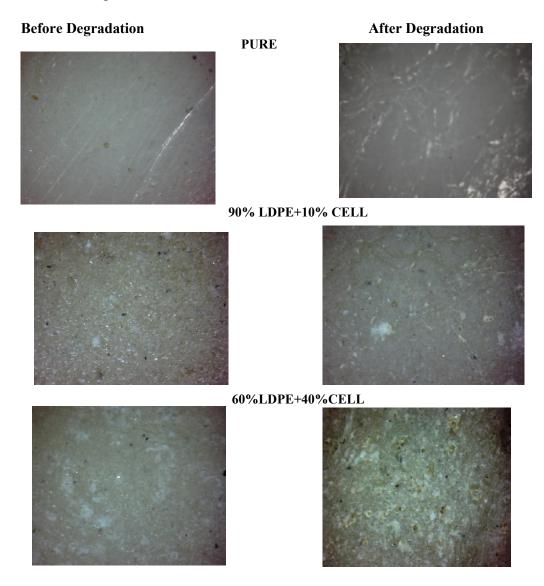


Fig.8: Effect of CELL on LDPE density

3.5 Morphology Test

The effect of degradation time on the biodegradability of the blends samples was obviously seen in the digital microscope(model AM4815T Dino-Lite Edge) made in (Kyoto Japan), with Magnification Rate (20x~220x).

It has been observed that the smooth surface of LDPE and the blend samples before burial test in soil. Inconstrat, the sample after burial in soil were rough and had alot of small pitting on the surface, while pure LDPE remain without change. The presence of pitting can be observed on the surfaces of the sample in soil. Increasing in cellulose contant led to higher surface pitting of the sample due to increase degradation. It can be considered that the change in physical appearance of the sample in the soil as evidence of biodegradation of this polymer in the landfills or natural environment. The result shows that the combination of hydrophobic LDPE with hydrophilic enhance the hydrophilicity and degradability of the overall polymer as show in figure 9.



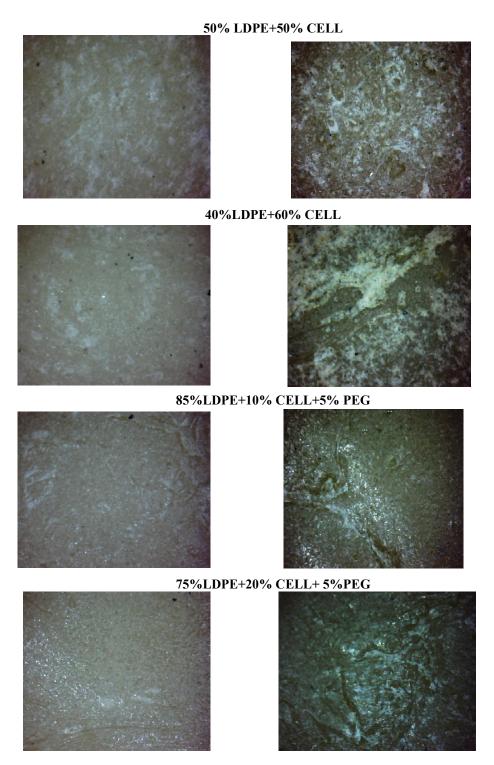


Fig. 9 Surface morphology of the LDPE polymer with different percentages of CELL.

3.6 Scan Electron Microscope (SEM) Test

Analytical Scanning Electron Microscope (SEM), model (JEOL 6400 F) which found in Razi Metallurgical Research Center (RMRC) in Tehran-Iran, used to examine the morphology of polymer blends. Figures 10to 12 show the SEM images of the samples. Figure11shows SEM image of the LDPE with cellulose, appearinadequate wetting of the fibre with the matrix, uneven aligning of the cellulose fibres and most probable poor adhesion between the filler and matrix this

reason gives poor mechanical properties. While addation PEG to the blends as show in figure 12, PEG works as the surface between the fiber and matrix increase the bonding strength; therefore observe a slight increase in mechanical properties.

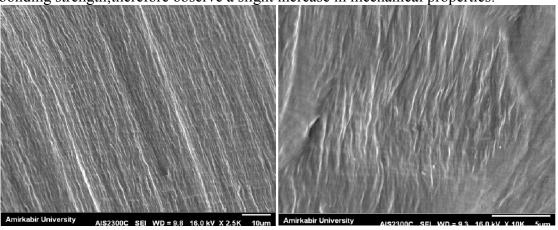


Figure 10: SEM Images of pure LDPE a, b Respectively.

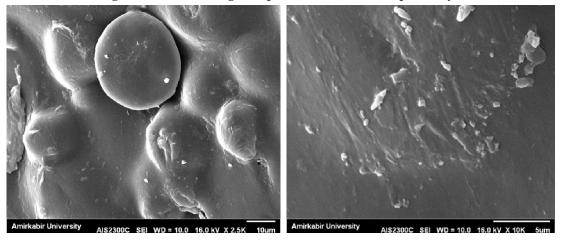


Figure 11: SEM Images of 60%LDPE+40%CELL a, b Respectively.

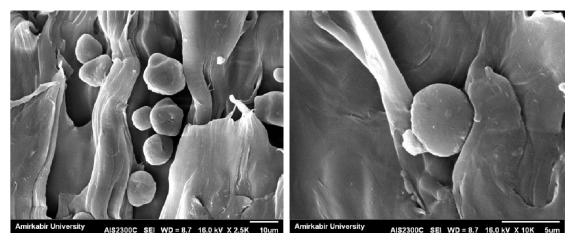


Figure 12: SEM Images of 55%LDPE+40%CELL+5%PEG a, b Respectively.

3.7 Degradation Tests:

3.7.1 Soil burial test

The biodegradation test experimental result for LDPE with cellulose blends show in figure 13. The dispersion and breakup of samples were noticed after about 40 days and increasing during burial time. It can be observed that higher percent of cellulose leading to higher degradation. These agreement with [Oldak and etal. 2005]. They found that with a very small percent of cellulose in polyethylene (PE) composite, it may not develope its biodegradability. The biodegradability in PE films will only have the pronounced effect if the blends contains 30% cellulose and above.

The effect addition of 5% PEG on the ability biodegradation of the LDPE/CELL blend as shown Figure 14, it was noted that increase the degradation rate generally compared with others samples not added her PEG as shown in Figure 13 so as to ability PEG to hydrolysis and biodegradation , also note the reduction degradation rate with the increase percentage of cellulose added due to formation secondary bonds between cellulose and PEG which reduces the ability cellulose and PEG on the degradation.

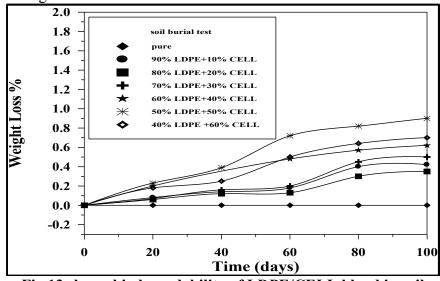


Fig.13 shows biodegradability of LDPE/CELL blend in soil SOIL BURIAL TEST PURE 85% LDPE+5% PEG+10% CELL 6 75% LDPE+5% PEG+20% CELL Weight Loss% 65% LDPE+5% PEG+30% CELL 55% LDPE+5% PEG+40% CELI 4 2 0 40 80 0 20 60 100 Time (days)

Fig.14 shows biodegradability of LDPE/CELL/PEG blend in soil

4. Conclusions

- 1.The tensile properties such as tensile strength and elongation at break of the LDPE/CELL blends decrease with increase (cell) percent, and the addation 5% PEG of the blendsled to improve values at certain percentage.
- 2. The soil burial test of LDPE/CELL blend showed that, the weight loss increases with increasing cellulose percent, while pure sample appear no weight loss and no surface deterioration in soil environments within the 100-day study period.
- 3. Adding 5% PEG of samples above observe increase the proportion of weight loss when placed in soil for 100 days with increase CELL percent added to LDPE.
- 4. The morphology test result appeared that the pitting is formed after burial in soil increases with increasing the time which show clearly propagation of degradation process.

Refrencess

- Behjat Tajeddin,2009"Preparation And Characterization Of Kenaf Cellulose Polyethylene Glycol- Polyethylene Biocomposites".
- Dmitri Shumigin, Elvira Tarasova, Andres Krumme, Pille MEIER,2011" **Rheological** and Mechanical Properties of Poly(lactic) Acid/Cellulose and LDPE/Cellulose Composites", ISSN 1392–1320 MATERIALS SCIENCE (MEDŽIAGOTYRA). Vol. 17, No. 1.
- Gulmine, J.V., a, P.R. Janissek b, H.M. Heise c, L. Akcelrud, 2002 " **Polyethylene characterization by FTIR"**, Polymer Testing 21, pp:557–563.
- Mark Jordi," **Degradation of Polyethylene by FTIR and High Temperature GPC**" Case Study, Report Number: JXXXX ,Jordi Labs LLC Confidential.
- Ołdak, D., H. Kaczmarek, T. Buffeteau, C. Sourisseau. 2005 "Photo- and Bio Degradation Processes in Polyethylene, Cellulose and their Blends Studied by ATR-FTIR and Raman Spectroscopies" Journal of Materials Science, , Volume 40, Issue 16, pp. 4189-4198.
- Samarasekara A., and E. Jayasuriya ,2011 "Synthesis of Biodegradable Polyolefins Based Polymer Composites Using Degradable Natural Materials" vol.18, International Forestry and Environment Symposium.