ARTICLE
JOURNAL OF UNIVERSITY OF BABYLON
For ENGINEERING SCIENCES (JUBES)
Vol.29, No.2.| 2021

Some Methodes for Measurements of Polymer Degradation: A Review

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Received:	31/9/2021	Accepted:	13/10/2021	Published:	22/10/2021

مجلة جامعة بابل للعلوم الهندسية مجلة جامعة بابل للعلوم الهندسية مجلة جامعة بابل للعلوم الهندسية

ISSN: 2616 - 9916

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Abstract

The purpose of this review article is to highlight the various approaches for measuring polymer degradation. Different tests (mechanical, thermal, rheological) give good evidence about polymer properties. From these tests, the effect of varying degradation factors on the general properties of the polymer can be estimated. Change in weight, thermal tests, dynamic mechanical analysis tests, structural tests, and the rheological test show the changes in the material behaviour before and after the effect of the different environmental factors.

Keywords: Degradation, DMA, DSC, TGA, FTIR, OIT.

Introduction

Polymer degradation is the reduction in the physical properties of a polymer, such as strength, caused by changes in its chemical composition. Polymers and particularly plastics are subject to degradation at all stages of their product life cycle, including during their production, use, disposal into the environment and recycling. The rate of this degradation varies significantly; biodegradation can take decades, whereas some industrial processes can completely decompose a polymer in hours[1].

Polymer degradation is a change in the properties of the polymer, such tensile strength, color, shape, and molecular weight, or of a polymer-based product under the influence of one or more environmental factors, such as heat, light, chemicals, or any other applied force. Degradation is often due to a change in the chemical and/or physical structure of the polymer chain, which in turn leads to a decrease in the molecular weight of the polymer. These changes may be undesirable, such as changes during use, or desirable, as in biodegradation or deliberately lowering the molecular weight of a polymer. Such changes occur primarily because of the effect of these factors on the chemical composition of the polymer. The susceptibility of a polymer to degradation depends on its structure. Epoxies and chains containing aromatic functionality are especially susceptible to ultraviolet degradation, while hydrocarbon-based polymers are susceptible to thermal degradation and are often not ideal for high temperature applications[2].

In general, the effects of heat, light, air and water are the most significant factors in the degradation of plastic polymers. The major chemical changes are oxidation and chain scission, leading to a reduction in the molecular weight and degree of polymerization of the polymer. These changes affect physical properties like strength, malleability, melt flow index, appearance and colour. The changes in properties are often termed "aging"[1].

Several types of degradation ocure in polymers during different conditions: 1) Degradation during processing such as thermal oxidation, thermal degradation and thermo-mechanical degradation, 2) In-service degradation such as chlorine-induced cracking, 3) Degradation in the environment such as photo-oxidation, hydrolysis, ozonolysis of rubbers and biological degradation, and 4) Degradation during recycling, remelting, thermal depolymerisation & pyrolysis and chemical depolymerization[2].

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The most crucial thing to carry out now a day is the degradation of different polymers. It poses a risk to both human health and to the environment. Some of the most consumed polymers by the population and difficult to degrade are PP, PVC, PVA, and low and high-density PE. Traditional gravimetric and respirometry techniques are currently used in research. They are easily adaptable to biodegradable polymeric materials. They're also ideal for biodegradable components made from polymer mixes. However, because recent polymer generations are more resistant to biodegradation, they cannot be used in this application. The information on various approaches indicated the best fit for individuals interested in evaluating polymer degradation under various environmental conditions and selecting an acceptable methodology for a specific blend of polymer and catalysts that aids in the degradation of the polymeric material[3]. Some of the methodes that can be used for measurement of the polymer degradation are listed:[4]

1. Weight Change
2. Thermal transitions (TGA, DSC)
3. Dynamic Mechanical Thermal Analysis (DMTA)
4. Change in molecular mass (GPC)
5. Fourier transform infrared spectroscopy FTIR
6. Structural Changes by SEM and AFM
7. Rheological properties using change in viscosity

1.Weight change

Weight change can be evaluated using soil burial or water immersion experiments, both of which are based on international standards for determining how materials rot and degrade. Biological activity is assessed in standardized test soil depending on various criteria throughout the test (chemical oxygen demand, temperature, soil respiration, soil moisture). Tests are conducted either in a realistic outdoor setting or in a controlled laboratory setting. Depending on the study issue or objective, the experiments are evaluated after a set amount of time has passed. Product stability, degradation rate (by morphological studies or tensile strength), exposure through decaying items (by chemical analysis or ecotoxicological tests), and environmental compatibility are all examples of things that can be evaluated. Standardized methods are used for each of these evaluations[3]. By evaluating the weight loss of the samples over time, soil burial could be studied. Every 20 days from the starting day, the weight loss was determined and can be calculated using Equation 1 [4]:

$$\%\ W_{loss} = [W_1 - W_2)/W_1] \times 100\% \ldots\ldots\ldots\ldots\ldots(1)$$

Where W1: sample mass before degradation in soil and W2:sample mass after degradation in soil.

Figure 1(a and b) is an example of the weight change method. Figure 1a depicts the weight loss (Wloss) of LDPE/PVA blends as a function of time in the soil environment. Pure LDPE samples did not show any weight loss or surface deterioration in soil environments during the 100-day study period; however, as the amount of PVA increased, weight losses increased (PVA content increases mass loss and improves degradation kinetics), which could be due to PVA's hydrophilic nature. The more PVA in a blend, the higher the moisture content, and thus the faster the deterioration. Due to the increased polarity and hydrophilicity of PEG when compared to LDPE/PVA blend, weight loss rises with the addition of PEG as shown in Figure 1b[5].

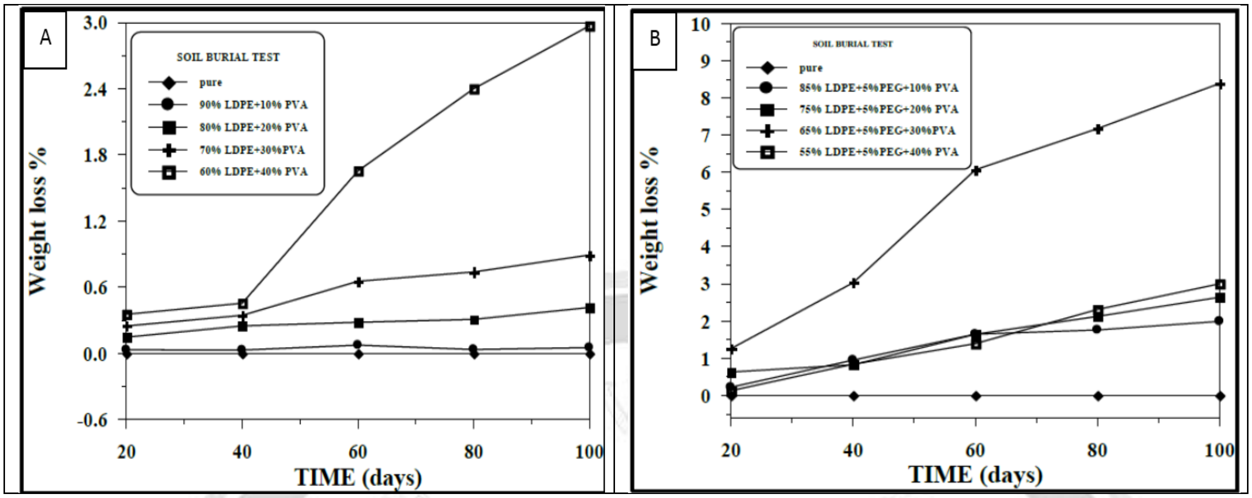

Figure 1: Biodegradability of LDPE/PVA in the soil environment, A) without PEG, B) with PEG[5]**.**

2. Thermal Degradation Techniques

TGA (thermogravimetric analysis) and DSC (differential scanning calorimetry) are two methodologies for evaluating polymer thermal degradation[6].

2.1. Thermogravimetry (TGA): is a method for determining the change in the mass of sample that has been exposed to a controlled temperature program. In recent years, isothermal and dynamic TGA has gained much attention for evaluating kinetic parameters in polymeric materials. Dynamic TGA is often used to explore the overall thermal degradation kinetics of polymers because it provides valuable data on exponential factor, the activation energy, and general reaction order, despite the fact that it cannot represent a specific thermal degradation process. Figure 3 shows changes in the slope and shape of the TGA curves as a result of different degradation parameters[7].

TG curve is classified in to six types on the basis of shape as shown in Figure 4[8].

1-Type 1: This type of curve exhibits no weight change throughout the complete temperature range used for analysis. The possible reason for this phenomenon may be due to the fact that the thermal stability of the sample is higher than the temperature range.

2-Type 2: This type of curve exhibits mass loss region which is then followed by a constant line. TG curves of type 2 results during certain processes such as drying where volatile compounds get evaporated and desorption.

3-Type 3: The TG curves which demonstrates single stage of weight loss or decomposition corresponds to Type 3.

4-Type 4: This type of curve can be exhibited by samples which undergo multi-stage decomposition process.

5-Type 5: This type of curve can be resulted when there is increment in weight for sample due to surface oxidation reactions that take place in a reacting atmosphere.

6-Type 6: This type of curve can be possible if there are multiple reactions with respect to rise in temperature. The increase in weight is due to the surface oxidation reaction whereas the decrement in weight with further rise in temperature corresponds to decomposition process of reaction products.

2.2. Differential Scanning Calorimetry: During the regulated temperature program in power-compensated DSC, the sample and reference material are kept at the same temperature. The temperature difference between the sample and the reference is then measured and compared to the set temperature. Both isothermal and dynamic thermogravimetric methods are employed. Dynamic DSC measures the Heat flow of a sample as a function of time while the temperature is continuously increased at a constant rate. Isothermogravimetric analysis (DSC) meausures the heat flow at constant temperatures as a function of time (stepwise change from one constant temperature to another). DSC can be used to investigate reaction heats, kinetics, heat capacities, phase transitions, thermal stability, sample composition and purity, critical points, and phase diagrams[9].

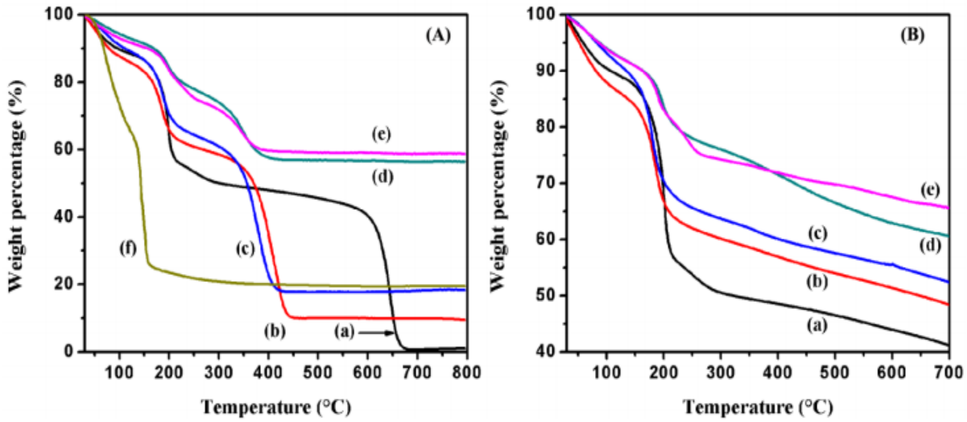

Figure 3: TGA curve with different atmospheres A) air , B) nitrogen.

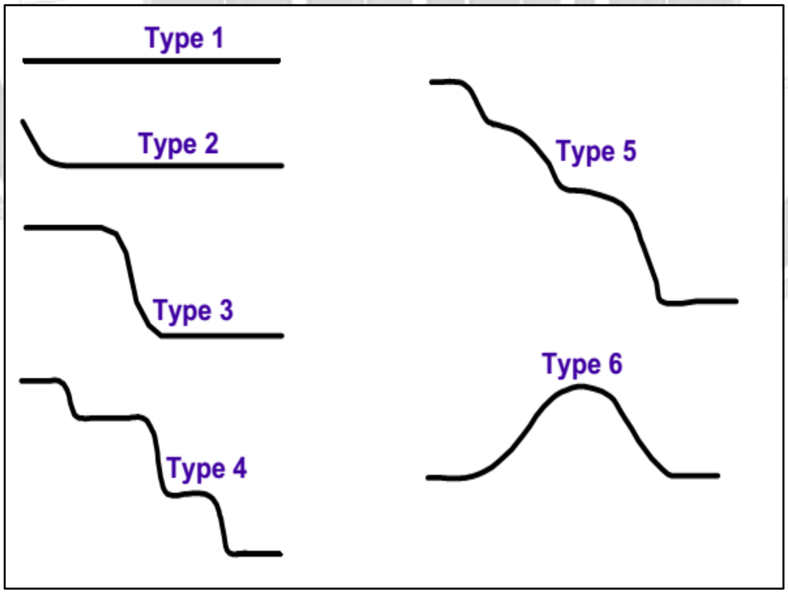

Figure 4: Classification of TGA curve[8].

The peak area is proportional to enthalpy shift after baseline adjustment to correlate the features captured with the thermal events happening in the sample.

$$\Delta H = K.A/m \ldots\ldots\ldots\ldots(2)$$

Where m is the sample mass and K is a constant. Thermal events in either an endothermic or exothermic direction (indicated on DSC curves) show as a deviation from the DSC baseline in the sample. Endothermic responses are frequently depicted as positive in DSC, that is, above the baseline[10].

Most solid polymers are formed by rapidly cooling to low temperatures (quenching) and are thus in a glassy state; heating them over Tg results in a glass transition, with a change in specific heat (Cp) but no change in enthalpy, resulting in no peak, simply discontinuity Figure 5.

2.2.1. The oxidation induction time / oxidative-induction time OIT

The oxidation induction time/oxidative-induction time test measures the time it takes for a polymer test sample exposed to oxygen to start oxidizing. Standard test procedures for OIT are ISO 11357-6 and ASTM D3895 Figure 6 [11].

An airtight sample chamber is typically needed when using differential scanning calorimetry to investigate sample stability to oxidation. It can be used to calculate a sample oxidative-induction time (OIT). These experiments are typically conducted isothermally (at a constant temperature) by modifying the sample atmosphere. First, an inert atmosphere, commonly nitrogen, is used to get the sample to the desired test temperature. The machine is then supplied with oxygen. Any oxidation is reported as a deviation from the baseline. The higher the OIT index (the gap between t1 and t2) as in Figure 6, the higher the resistance of material to oxidation. This type of analysis can be used to assess material or compound stability and ideal storage conditions[12].

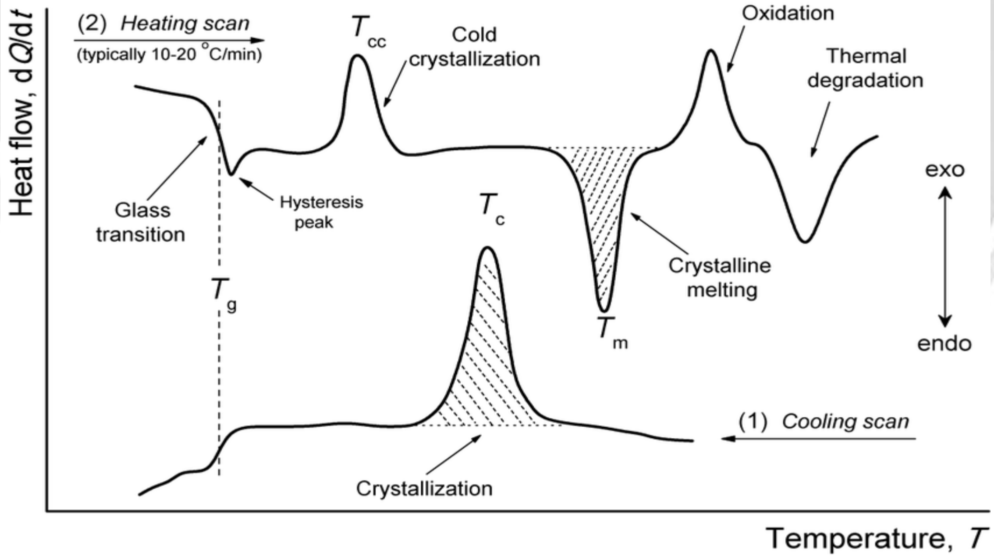

Figure 5: Thermal transitions in DSC curve[13]**.**

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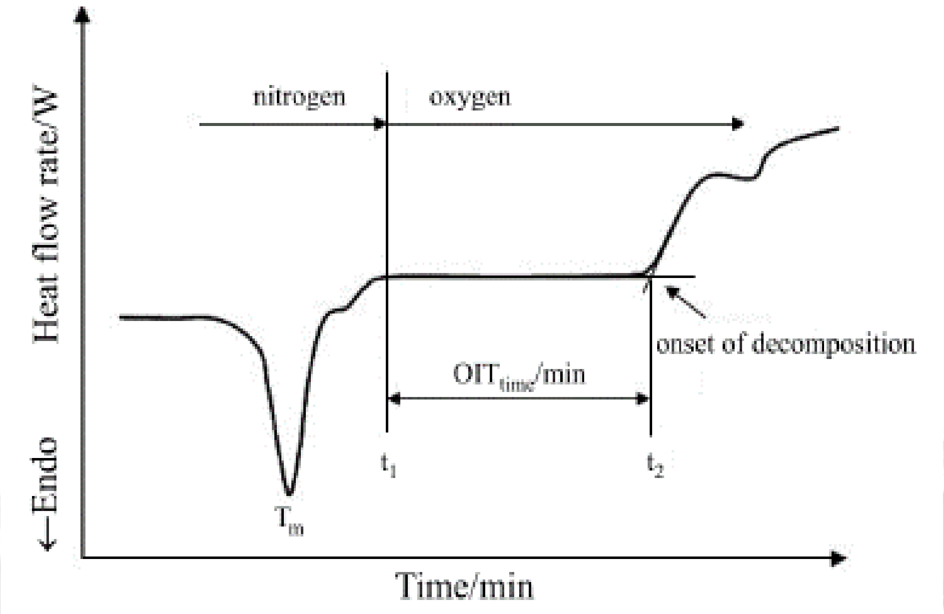

Figure 6: Principle measurements of OIT[14].

3. Dynamic Mechanical Analysis (DMA)

In the polymer and rubber industries, DMA or DMTA is a common thermal testing procedure. Materials degradation is studied using this method. It entails placing an oscillatory force or deformation on material and seeing how it reacts[15]. Because sinusoidal stress is applied, modulus can be expressed as;

- The in-phase storage modulus (E ') and the out-of-phase loss modulus (E").
- Storage modulus (E ') measures material elastic response and measures the energy stored. Loss modulus (E") measures viscous material response, which measures the dissipated energy as heat.

Polymers can respond to motion energy in two ways: elastically, which is required for form restitution, and viscously, which is required for dispersing mechanical energy and preventing breaking[16].

The following are some of the major DMA research areas that meet the demand for precise and rapid assessment of polymer mechanical properties[17]:

1. Determining the glass transition temperatures (Tg) of polymers[18].
2. Monitoring changes in the elastic or (shear) modulus as a function of frequency when exposed to various environmental factors.
3. Determining what constitutes "damping" behaviour: Internal motion is used to dissipate mechanical energy (loss modulus, tan delta)
4. Comparing the mechanical properties of several polymers, such as quality, failure analysis, new material qualification, or the same polymer after being exposed to various environmental conditions.
5. Polymer blends or copolymers are being studied for phase separation.

6. Physical ageing, crosslinking, and post-cure effects on mechanical properties and Tg.
7. Using transient measurements, assess creep, stress relaxation, and stress-strain characteristics.

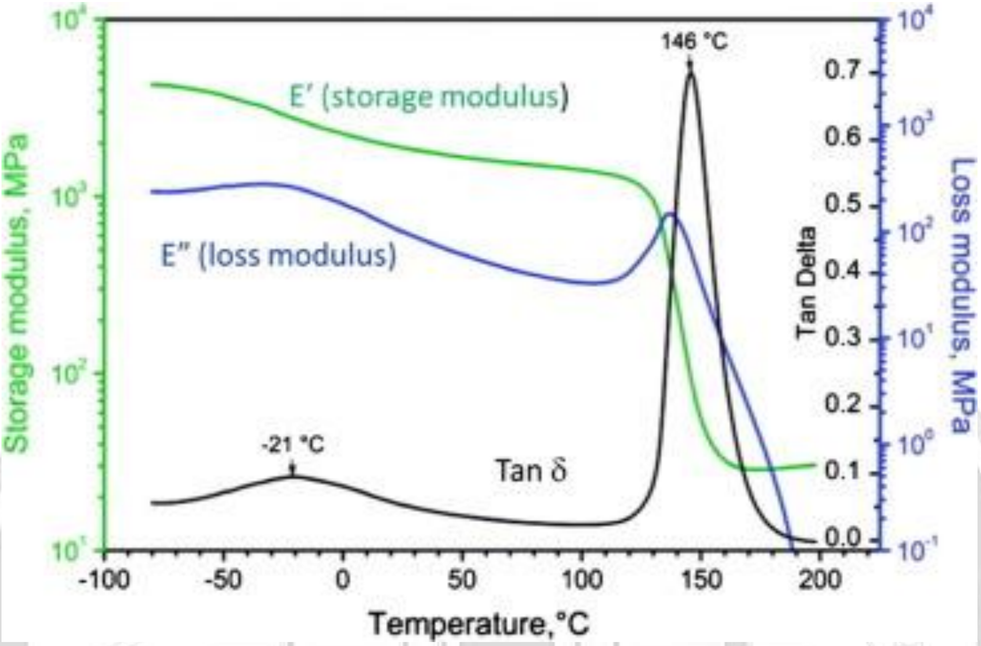

Figure 7: Relationship between E', E" and tan delta Vs temperature in the DMA[18].

Figure 7 shows the difference in tan delta and storage modulus during heating the sample as a function of frequency. As a result of changing frequency with increasing temperature, it's found that the modulus increases (material stiffness increase) when compared with standered sample (not exposed to degradation factors) due to heat or light and tan delta decreases for the same results [19] and [17].

4. Molecular mass change (Gel permeation chromatography GPC)

The average molecular weight distribution of a polymer sample is determined using GPC technique, which also known as Size Exclusion Chromatography (SEC). Using the right detection and analysis methods, it is possible to get qualitative data on long chain branching or to identify the composition distribution of copolymers. GPC or SEC separates polymers according to their size or hydrodynamic radius, as the name suggests. A modest volume of polymer solution (0.0l-0.6 percent) is injected into a succession of porous bead-filled columns to achieve this (100-400 micro L). Smaller molecules can pass the pores more easily and are held longer than larger molecules, which proceed down the columns and elute faster. The output of the columns is connected to one or more detectors[20].

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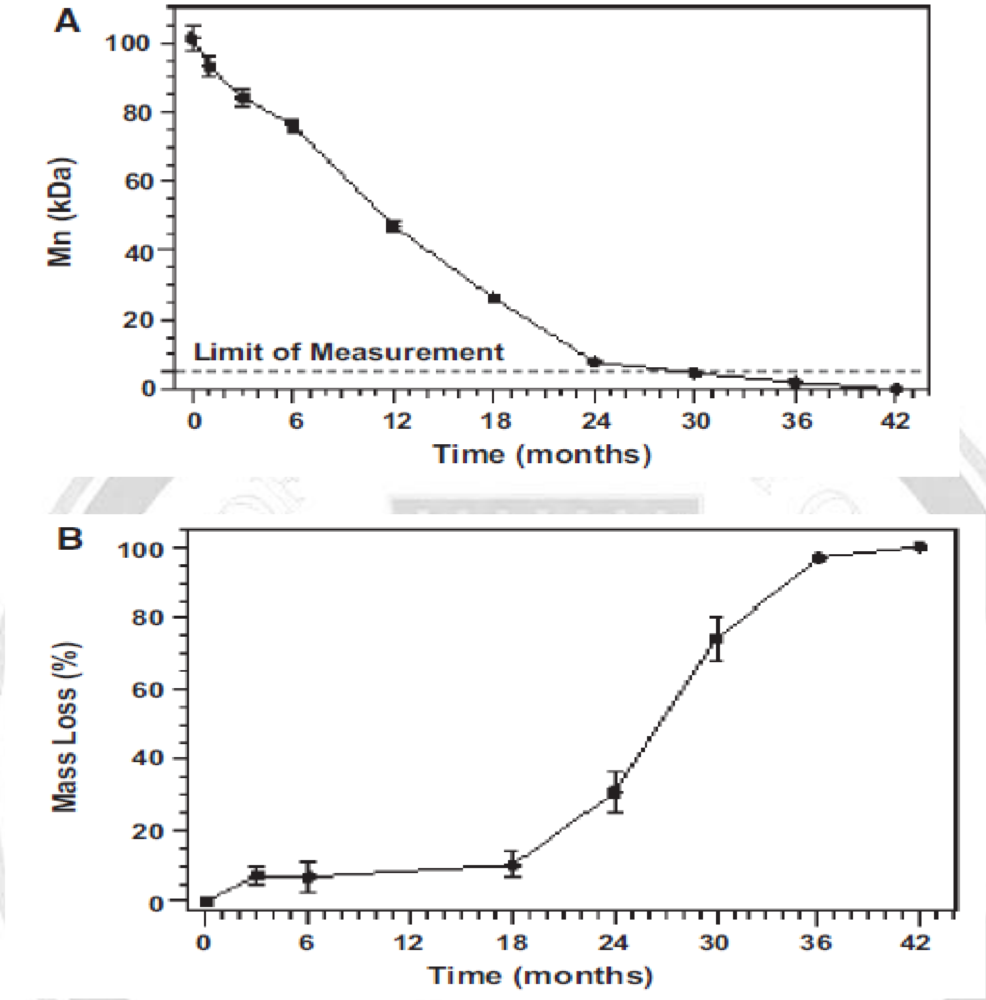

Figure 8: Molecular weight and mass loss change using Gel permeation chromatography (GPC)[21]**.**

Figure 8 illustrates the use of Gel permeation chromatography (GPC) to evaluate the degradation of Absorb everolimus-eluting bioresorbable vascular scaffolds (Absorb). A: the in vivo degradation of polymer of absorb over time. The lower limit of measurement for the number-average molecular weight (Mn) is 4.7 kDa. B: The in vivo mass loss of polymer of Absorb over time. The limit of detection for the mass loss is 0.1 mg/mL[21].

5. FTIR Technique

A new chemometric technique based on Beer's law was developed to assess the breakdown of individual polymers during biodegradation, which analyzes peak ratios from Fourier transform infrared spectra of neat polymers and their composite plastics. Without any prior calibration against existing composites, the method detects polymer concentrations and weight losses directly. This method, unlike standard chemometric methods, does not require many tests as the number of samples increased. Only two observations, one before and one after biodegradation, are necessary after the tidy polymer spectra are discovered. The method allows for computer-assisted selection of analytical infrared wavelengths among all possible wavelength combinations, which could be a significant advancement[22].

The results of FTIR to investigate the mechanism of the degradation process of the decomposed matrix polymer (DMP) obtained at 190 °C are displayed in Figure 9. For the best comparability, the internal standard used to standardize the spectra was a peak at 1607 cm-1. Peak intensities at 1741 cm-1 (C=O in the ester bond) and 1148 cm-1 (C–O–C in the ester bond) decreased as the ester bond deteriorated, indicating ester bond cleavage[23].

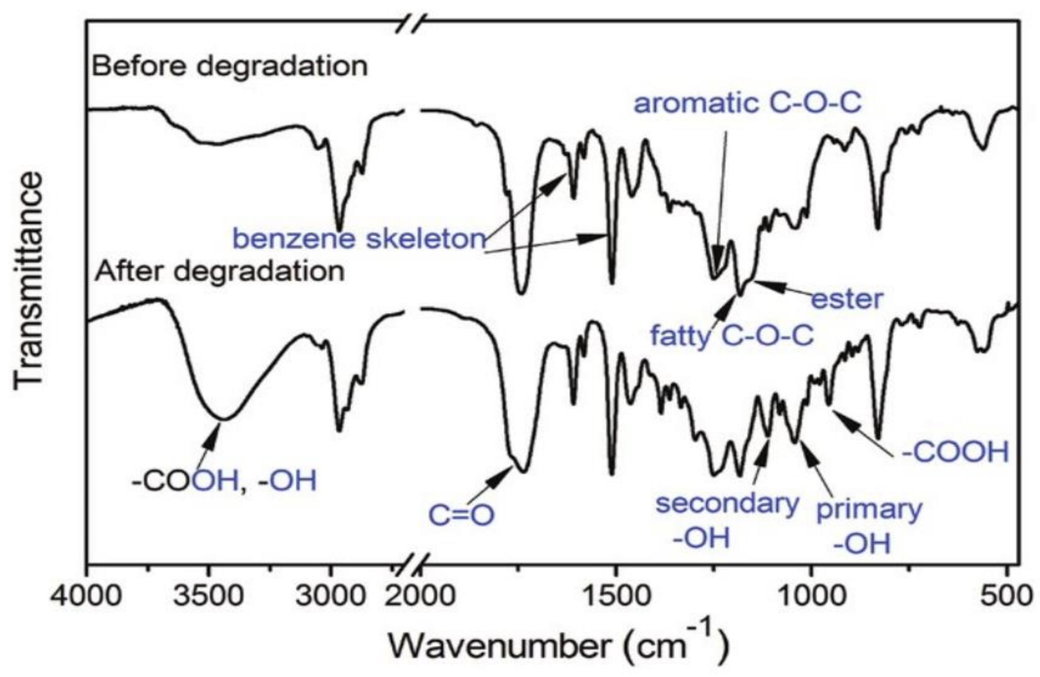

Figure 9: FTIR for the DMP before and after degradation[23].

5.1. Thermogravimetry-Fourier transform infrared spectroscopy TG-FTIR

In a controlled environment, thermogravimetric analysis (TGA) is used to measure the change in weight of a sample as a function of temperature or time. FTIR was used successfully in the identification of gases. Combining these two approaches allows for comprehensive material characterization in terms of decomposition mechanisms and thermal stability[24].

Figure 10 shows the evolution of CO2 from the FTIR chemigram was plotted against the weight loss of catalyst from the TGA Figure 10; Two CO2 peaks were observed in the Chemigram profile; this indicates that two forms of carbon were formed. Previous studies indicate that the CO2 generated at the lower temperature (360 °C) was due to coking on the active sites of the surface of the catalyst while the other type of carbon formed (544 °C) was pseudo-graphitic in structure and contains polyaromatic compounds. These are most likely formed in and on the catalyst support or in some cases at the interface between the catalyst supports and the active sites[25].

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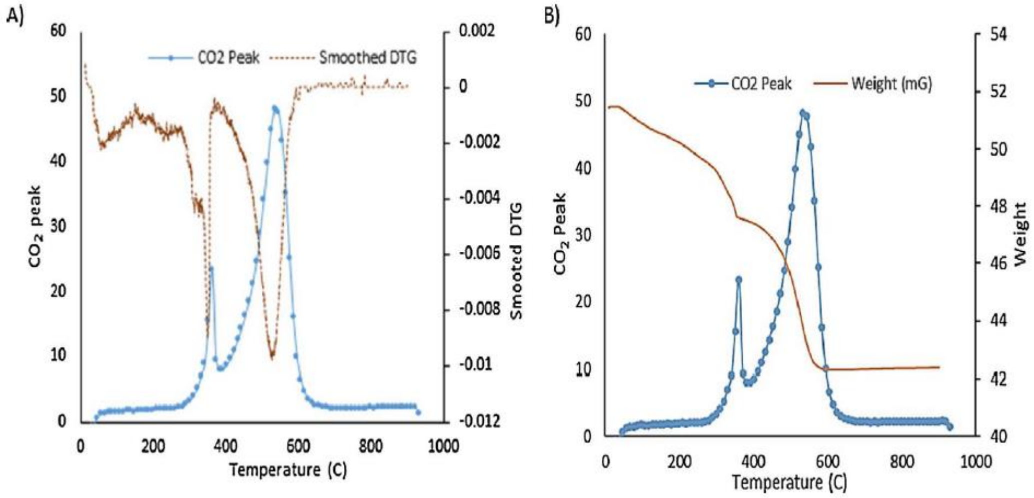


Figure 10: TGA/FTIR analysis of used CLSR catalyst B, A) CO$_2$ against smoothed DTG B) CO$_2$ against weight loss of catalyst[25]**.**

5.2. Thermogravimetry-Mass spectroscopy TG-MS

When thermal analysis (TA) is combined with gas-analytical techniques, the chances of accurately interpreting the mechanism of thermally driven processes involving the generation of gaseous species are greatly increased[26]. The great sensitivity and resolution of TG-MS allow for the detection of extremely low concentrations of generated gases, as well as overlapping weight losses that can be qualitatively interpenetrated. This approach consequently gives qualitative information about the evolved gases during polymer degradation that would otherwise be unavailable in TG-only tests. As a result, this technique is used to characterize the structural properties of homopolymers, copolymers, polymeric blends, and composites, as well as to deflect monomeric residuals, solvents, additives, and harmful degradation products[27].

6.Structural changes

6.1. Scanning Electron Microscope SEM

SEM is an electron microscope that scans a sample with a focused beam of electrons to produce pictures. It is one of the most powerful tools for inspecting and analyzing microstructure morphology and chemical composition characterizations currently accessible. When electrons contact with atoms in a sample, they generate a variety of signals that may be detected and contain information about the surface topography and composition of the sample [28]. Figure 11 show how SEM is used to study microstructure of materials when exposed to varios degradation factors.

The degradation of PLA and its composites with starch in compost and soil also indicates the process catalysed by enzymes in the case of composites. SEM micrographs as in Figure 11 showed changes in surface morphology (cracks) of PLA and its composites after degradation in compost and soil for 14 days. (Bio)degradation occurred for all polymer samples tested, but more changes were observed in PLA/starch (50/50) composite than on pure PLA samples. This means that the addition of starch to PLA resulted in their greater biodegradability[29].

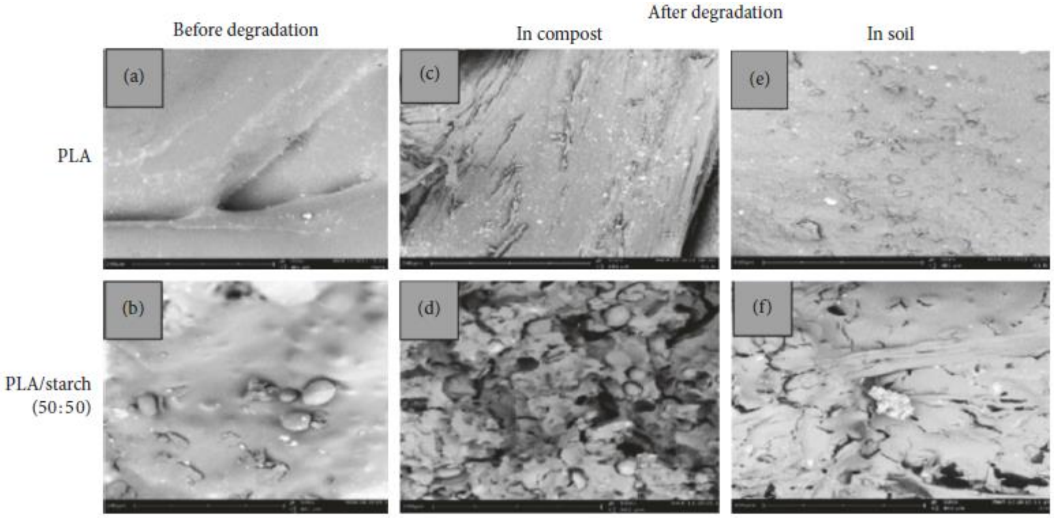
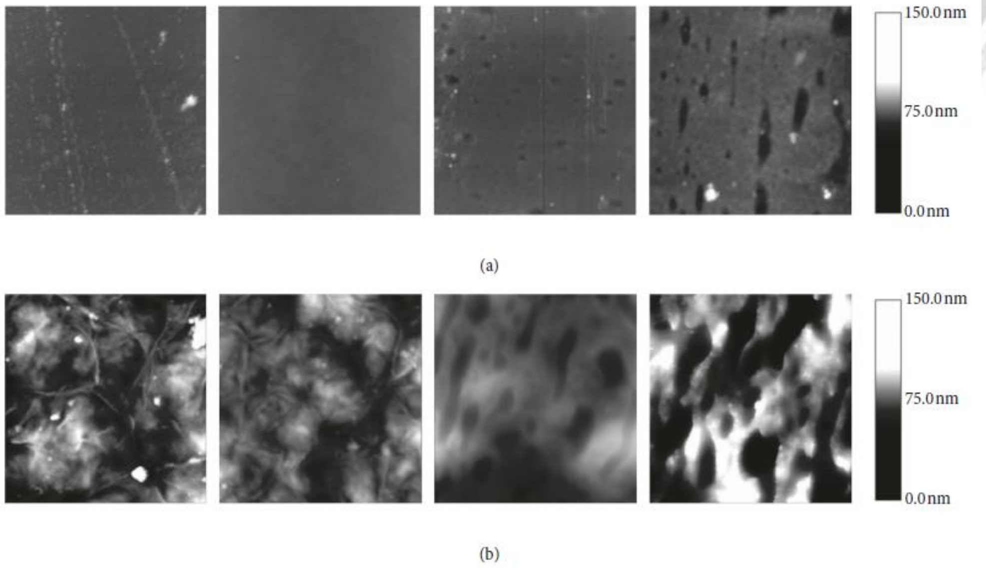


Figure 11: PLA and PLA/starch composite SEM images before and after degradation[29].

6.2. Atomic force microscopy (AFM)

Because the phase response of the cantilever is sensitive to surface properties, such as friction, adhesiveness, and resistance, the phase imaging mode is actively involved in mapping surface heterogeneity of hybrid materials, blends, and composites without requiring sample preparation or a vacuum setting. AFM is a precies technology to asses the changes in surface properties when the material subjected to different degradation enviroments, Figure 12 show the difference in surface of PLA before and after degradation.

The degree of erosion of the polymeric material was determined by AFM as shown by Figure 12 and it is found that ther erosion rate strongly dependent on the blend miscibility of the two components. the poor miscibility of the blend with the largest pit domain before degradation promoted greater roughness of the surface during degradation[29].



(a)

(b)

Figure 12: AFM images of PLA, (a) before and (b) after the degradation[29].

7. Rheological properties

Different environmental factors affect the properties of the polymer and blend and cause degradation. One method to measure the effect of environmental factors on polymer degradation is rheological properties. Rheological characteristics can be used to measure polymer degradation by testing polymers and polymer blends with capillary viscometer or melt flow index in the case of solid polymers or by using cone-plate or plate -plate in case of liquid polymers. The change in viscosity with shear rate can be plotted for both cases, before and after the degradation environment[30] and [31].

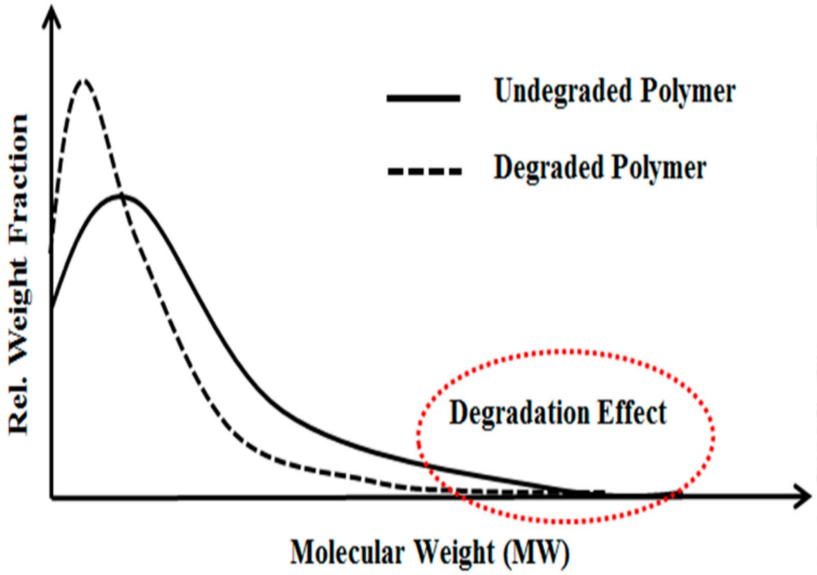


Figure 14: mechanical degradation effect on the molecular weight distribution of polymer (MW)[32].

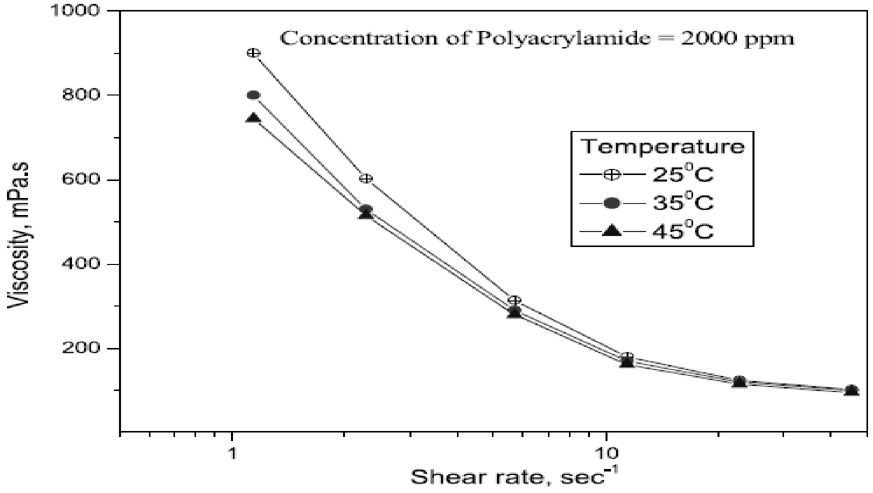


Figure 15: Effect of temperature on viscosity of polymer solution[33].

Conclusion

The paper discussed several methods for measuring the degradation of polymers. Since there are several factors that affect polymer degradation, there are several test methods. TGA and DSC show the change in thermal transitions during heating the material, FTIR shows changes in band values (appearance or disappearance), structural test (SEM, AFM) shows the changes in composition, surface roughness, and rheological tests shows the difference in viscosity.

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مراجعة لطرق قياس تحلل البوليمرات

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الخلاصة:

ان الغرض من إعداد ورقة المراجعة هو تسليط الضوء على الطرق التي يمكن استخدامها لقياس تحلل البوليمر. تقدم الاختبارات المختلفة (الميكانيكية والحرارية والريولوجية) دليلًا جيدًا على خصائص البوليمر، بحيث يمكننا من هذه الاختبارات تقدير تأثير عوامل التحلل المختلفة على الخصائص العامة للبوليمر. يظهر التغير في الوزن والاختبارات الحرارية واختبارات التحليل الميكانيكي الديناميكي والاختبار التركيبي والاختبار الريولوجي التغير في سلوك المواد قبل وبعد تأثير العوامل البيئية.

الكلمات الدالة: التحلل، التحليل الميكانيكي الديناميكي DMA، مسعر المسح التفاضلي DSC، التحليل الوزني الحراري TGA، FTIR، وقت بداية التاكسد OIT.