INFLUENCES OF ULTRASONIC BATH SUBSTANCES ON MECHANICAL PROPERTIES OF DEGRADED POLYPROPYLENE (PP) AND LOW-DENSITY POLYETHYLENE (LDPE)

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ABSTRACT

Varieties of plastics waste are commonly ended in landfill, ocean and polluting water bodies. Recycling efforts of these plastics waste in using high temperature often resulted in low quality of recycled resins. Ultrasonic process has been applied in sample treatment for mass spectrometry-based techniques and synthesis to obtain homogeneous distribution of monomers in polymeric materials. Ultrasonic induced vibration and splitting of polymer chain shown by declining physical properties. Low temperature (30 - 50 ± 2ºC) ultrasonic range of 2 main plastics, polypropylene (PP) and low-density polyethylene (LDPE) in dumbbell shape were evaluated on 3 different ultrasonic media for 30 – 150 minutes. Ultrasonic bath of concerned included water (W), water with toluene (1% v/v)(WT) and water with aluminum strap (1% w/v)(WA). The samples were prepared and mechanically tested using ASTM D638, ASTM D790 and ASTM D256 methods. The present of toluene and aluminum strap accelerated degradation of both PP at 30 and 40ºC at 30 min of reaction. Prolong reaction time had shown declining mechanical properties of PP and LDPE. However, at 50ºC different trend of LDPE degradation was observed. Organic solvent had assisted LDPE degradation but the present of aluminum strap increased mechanical properties of LDPE prolong reaction time. The findings confirmed on ultrasonic bath brought changes on the mechanical properties of PP and LDPE signifying degradation or enhancement of the material. Degradation of LDPE and PP responded differently towards ultrasonic temperature and treatment time. Low temperature ultrasonic is promising for degradation of PP and LDPE with the present of toluene and aluminum strap.

Keywords: Degradation trend, low temperature ultrasonic, mechanical properties.

INTRODUCTION

Petroleum-based polymer materials play an extremely important role in modern civilization. Most of technological products and devices are made of polymers. The most common polymer materials used are polypropylene (PP) and Low-density polyethylene (LDPE). Abundance of PP and LDPE post-consumer waste however causing environmental problems in the ecosystem due to their resilience properties against degradation and proliferation in industry. The issue of plastics pollution has threatening the global ecology severely. Both terrestrial and marine sources are continually fed the plastics pollution stream either purposefully or illegally (inappropriate dumping of domestic and industrial waste) [1]. Efforts on plastics recycling and diversion of plastics from landfill are extremely important to extend the life cycle of plastic. Common method of recycling applied high heat to melt them and forming recycled low quality pellets. On the other hand, conversion of these plastics into variable degraded products can be classified into a few degradation methods such as super critical water [2], ultrasonic [3][4], pyrolysis [5][6] and microbial degradation [7]. The concern was on the recoverable of repeating units and certain properties to suit further applications.

Ultrasonication has become a tool in polymer research with three different aims. Ultrasonication has long been used in the synthesis of polymers to obtain a homogeneous distribution of the monomers, also used to study polymer degradation mechanisms. And recent approach deals with the utilization of ultrasonication for fast sample treatment prior to the characterization of polymers by mass spectrometry-based techniques. Ultrasonic degradation occurs on application of sound at certain frequencies that can induce polymer chain to vibrate and split [8]. All forms of degradation represent chemical reactions that result in molecular structural changes and accompanied by decline in physical properties. Only ultrasonic frequency from 20kHz to a few MHz can cause (acoustic) cavitation and at lower frequency, more violent cavitations will be produced, resulting in higher localized temperatures and pressure [9][10]. In the present study, ultrasonication is in context of ultrasonic degradation induced by sound waves to degrade a specimen. Sound waves carry energy along as they travel across a medium and concentrating diffused energy.

Degradation through ultrasonic extended beyond specimen’s surface described as three layers of cavitation bubbles [11] forming microbubles of water dissociate into hydroxyl (OH^-) and hydrogen atom (H^+) radicals [12]. The radicals attack polymer carbon-carbon chain (skeletal) preferentially at the middle of the chain with more physical and chemical effects in lower ultrasonic temperature. In this temperature range, ability of ultrasonic mechanism to generate more free radicals and actively attacking the mid-point of the polymer skeletal chain is intensive. Hence, ultrasonic energy can also be used to influence polymer properties to create or alter specific characteristics, termed as controlled ultrasonic degradation. Ultrasonic degradation of PP and LDPE hydrophobic plastics was reported to be limited in excess solvent concentration and operating temperature increasing the viscosity [4]. Thus the needs on investigating low temperature and minimum amount of solvent are crucial for further characterization of the mechanical properties of degraded PP and LDPE in ultrasonic degradation.

METHODOLOGY

Dumbell shape samples preparation

Injection grade low density polyethylene (LDPE) pellets/PETLIN® LDPE and polypropylene (PP) pellets/TITANPRO® were purchased from local suppliers. The pellets were kept at room temperature at least 24 h before injection molding. The Battenfeld BA600/200 CDC injection molding machine was pre-set at four temperatures setting; T_0 220ºC, T_1 223ºC, T_2 210ºC and T_3 180ºC producing dumbbell and rectangular shapes specimen for ultrasonic treatment according to the international standards, ASTM D3641-15. The samples were kept at room temperature prior used following ASTM D618-13 in ensuring substantial equilibrium under normal conditions of humidity and temperature in stipulated period. Furthermore, they were kept away from sunlight and fluorescent light as potential degradation sources.

Ultrasonic degradation conditions

Elma Transsonic Ultrasonic Cleaner T820/H was used to perform three ultrasonic degradation bath conditions. Ultrasonic bath of 3 liter distilled waters with following conditions and labels; water (W), with toluene (WT) (1% v/v) and water with aluminum strap (WA)(1% w/v) were kept constant for all degradation processes within stipulated temperatures and duration. Ultrasonic equipment was operated with 35 kHz ultrasonic frequency and fixed 1120 W of input power. Triplicates dumbbell and rectangular shape samples of PP and LDPE were placed in ultrasonic bath from 30 to 50 ± 2ºC for 0 – 150 min with 30 min intervals. The ultrasonic internal compartment was rinsed with distilled water and cleaned before proceed with following degradation conditions.

Degraded samples conditioning

Pre-weighted degraded samples were put in desicator overnight to remove residual moisture. They were further conditioned at standard laboratory atmosphere for a minimum of 40 h with adequate circulation on all its individual sides prior to mechanical testing according to ASTM D618-13 standard. All samples were then kept away from sunlight and fluorescent light as potential degradation sources prior mechanical testing.

Mechanical testing methods

To assess the mechanical properties, all conditioned degraded samples were subjected to impact test (ASTM D256), flexural test (ASTM D790) and tensile test (ASTM D638-13). Impact test was performed using Ray Ran Universal Impact Tester with hammer weight of 1.189 kg, impact velocity of 2.9 m/s and energy of 5 Joules. The test temperature was 27˚C. The 3 mm thickness samples were prepared by cutting the rectangular samples to 80 mm length using a cutter. Flexural tester, Lloyd R 30K Plus was used for flexural testing at standard laboratory atmosphere. Each sample was marked with 25 mm gauge length. Pendulum speed in the test was maintained at 10 mm/min crosshead rate and maximum deflection was 30 mm and placed for three points bending. The tensile stress test was performed using tensile tester, Instron 3366 at standard laboratory atmosphere of 23 ± 2 °C and 50 ± 5% relative humidity. The tensile stress was conducted at 40 mm/min of crosshead speed and the grip distance was 101.6 mm. Each sample was marked at midpoint of the total length and with 30 mm gauge length. The stress break and stress at maximum load transmitted via the equipment sensor were calculated by the software and displayed through the monitor. The tensile modulus is the ratio of stress break to strain, calculated in MPa. All obtained data were recorded in the software-oriented computer attached each testing equipments.

RESULTS AND DISCUSSION

Characteristics of PP and LDPE

The characteristics of pristine PP and LDPE are tabulated in Table 1. Relative toughness or impact toughness of PP is much lower than LDPE and most likely to fracture under high speed stress. The impact values of PP and LDPE are is 3.16 ± 0.03 kJ/m^2 compared to LDPE at 19.31 ± 0.12 kJ/m^2. LDPE has higher hardness compared to PP because it can withstand higher impact energy. Based on the observation during the testing, PP sample is easily broken into two parts at the v-notch area compared to LDPE. PP was not able to absorb the amount energy of the pendulum hitting the sample compared to amount of energy of LDPE. From this condition, PP sample is more rigid and less flexible compared to LDPE, which lead to fractured.

On the other hand, tensile strength of PP was higher than LDPE with 19.346 ± 1.776 MPa and 10.723 ± 0.315 MPa respectively. This indicate the ductility of PP than LDPE thus is has higher resistance to breaking under stress. Further more, PP also has higher young modulus compared to LDPE which is more less rigid, 1450.99 ± 1.65 and 185.13 ± 4.54 Mpa respectively. In term of stiffness, PP has more stiffness than LDPE. Basically, chemical structure of PP is asymmetrical and Polyethylene (PE) is more linear. This resulted in high rigidness of PP and more pressure is needed to bend the sample in flexural test. Bending was recorded at maximum load until failure state.

Table 1. Mechanical characteristics of pristine PP and LDPE

Parameter	Units	PP	LDPE
Impact value	kJ/m^2	3.16 ± 0.03	19.31 ± 0.12
Tensile strength	MPa	19.346 ± 1.776	10.723 ± 0.315
Young Modulus at bending	MPa	1450.99 ± 1.65	185.13 ± 4.54
Maximum bending stress at maximum load	MPa	40.089 ± 0.28	6.38 ± 0.03

[a] Values represents the average ± SD of 3-5 samples.

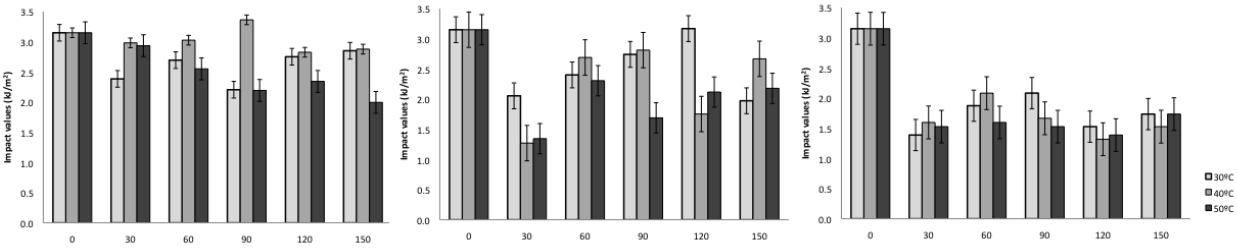
The bending stress make the crystalline part to withstand the uniform stress that applied to the sample. As the load is applied on the sample, the stress that produced from the process of bending. Both PP and LDPE not have same trending due to different molecular orientation. The relationship between material and bending stress at maximum load for PP is higher than LDPE which are 40.089 ± 0.28 and 6.38 ± 0.03 respectively. From this value, LDPE can handle more stress that applied to it compared to PP because PP is a rigid material and has more crystalline part in molecular orientation of the material. These mechanical properties of pristine PP and LDPE samples were also affected by the samples preparation and the additives.

Effect of ultrasonic temperatures and baths substances on impact values of PP and LDPE

Figure 1 shows the impact values (kJ/m^2) of degraded PP samples at different ultrasonic temperatures from 30 - 50ºC. In W bath ultrasonic, generally PP impact values are in fluctuating trend adjusting to degradation process that taking place. The value drops the lowest to 2.21 kJ/m^2 at 30 minutes in 30˚C and rising the highest to 2.85 kJ/m^2. Prolong of ultrasonic at 150 min reduced the impact values of 9.8% from pristine PP at 30˚C. Ultrasonic temperature of 40˚C was not favorable for PP degradation indicated by minimal drop of impact values throughout 150 min duration. However, interesting trend was observed at 90 min where the impact value was increased to 3.36 kJ/m^2, which was 6% higher than pristine PP. However, prolong of the ultrasonic time, shown degradation of PP samples. At higher ultrasonic temperature, 50˚C, decreasing impact values trend can be clearly observed with an increased value of 2.34 kJ/m^2 at 120 min. However, prolong to 150 min, the values decreased to the lowest of 1.99 kJ/m^2 indicating degradation of PP.

Present of toluene and aluminum strap in the ultrasonic bath increased the degradation rate of PP. Toluene has affected the degradation in fluctuated manners and prolong time to 120 min at 30˚C shown an increased of impact values to 3.17 kJ/m^2. Increment of ultrasonic temperature assisted degradation of PP in WT bath indicated further scission of PP backbone in homogenous state to lowest value of 1.32 kJ/m^2. High degradation temperature needs shorter reaction time in WT bath to achieve more degradation rate. Present of aluminum in WA bath shown fluctuation with high degradation rate of PP. Ultrasonic temperature of WA do not seem to drastically affect the degradation rate thus the percentage of aluminum (w/v) might accelerate the degradation at low ultrasonic temperature. A minimal impact values of 1.73 kJ/m^2 increment was recorded at 50˚C, 150 min. Interestingly, the lowest values of 1.32 kJ/m^2 was recorded slightly before the increment supporting the non-random hydro-chemical processes took place in the bath.

PP degradation trend based on toughness properties in three different ultrasonic baths could be explained by the act of cavitation towards the plastic. High rigidity and less flexible PP was prone to ultrasonic degradation through non-random process attacking the mid-point of backbone chain produced fragmentation [Desai 2008a] thus weaken the plastic. Fluctuated impact values indicated both hydro-mechanical process of ultrasonic degradation occurring at the same time lead to further degradation of PP at 40 and 50˚C. On the other hand, steady constant degradation only observed at lowest ultrasonic temperature of 30˚C clearly proved non-random chain scission of PP.



(W) (WT) (WA)

Figure 1. Impact values (kJ/m^2) of degraded PP samples at different ultrasonic temperatures and baths.

Effect of ultrasonic degradation on LDPE impact values with various bath conditions was shown in Figure 2. LDPE that is a flexible plastic responded well to the ultrasonic degradation. Gradual decreased impact values was recorded for ultrasonic bath at 30ºC from 30 – 90 min reaction time with the lowest value of 15.25 kJ/m^2 from 20.81 kJ/m^2. Prolong of reaction to 120 – 150 min indicated increased to 19.25 kJ/m^2 impact values. On the other hand, at 40ºC, considerable fluctuation impact values can be observed with the lowest value of 14.76 kJ/m^2 and highest of 22.41 kJ/m^2 at 90 min. Vigorous degradation trend of LDPE degradation in W bath of 29% and increment of 7.7% from pristine LDPE. Ultrasonic baths of WT and WA shown similar impact values trend of W bath for LDPE at 30ºC. At 40ºC, fluctuated degradation trend indicated by the highest impact value of 24.56 kJ/m^2 as early as 30 min of reaction time. WA bath seem to has less effect on LDPE degradation but recorded a few increment of within 4 – 7% from pristine LDPE with impact values of 21.67 and 22.32 kJ/m^2 respectively at 40 - 50ºC. On the other hand the lowest impact value at 50ºC was recorded with decrement of 27.5% from pristine LDPE.

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Effect of ultrasonic temperatures and baths substances on tensile strength of PP and LDPE

Figure 3 shows the changes in tensile strength of PP degraded samples within 30 – 150 min of different ultrasonic baths. Prolong reaction time in W bath to 90 min shown an increased of tensile strength and gradually decreased at 40˚C with the lowest of 18.547 MPa at 60 min and more fluctuated trend at 50˚C. On the other hand, for 30˚C, tensile strength gradually increasing from 19.356 to 21.973 MPa. The present of toluene and aluminum had impact on the PP as indicated by the significant increased of tensile strength at 50˚C for both WT and WA baths. Similar trend of tensile strength had been observed for W bath at 30˚C as compared to higher temperature of 50˚C for WT and WA bath. Shorter reaction time were enough for degradation of PP in WT and WA bath within 30 min with 32% decreased in tensile strength. PP has good chain networking to each other thus longer degradation time is needed to degrade in W bath. While in the WT and WA, both toluene and aluminum affected the PP chain reaction thus resulted in degradation of the tensile strength.

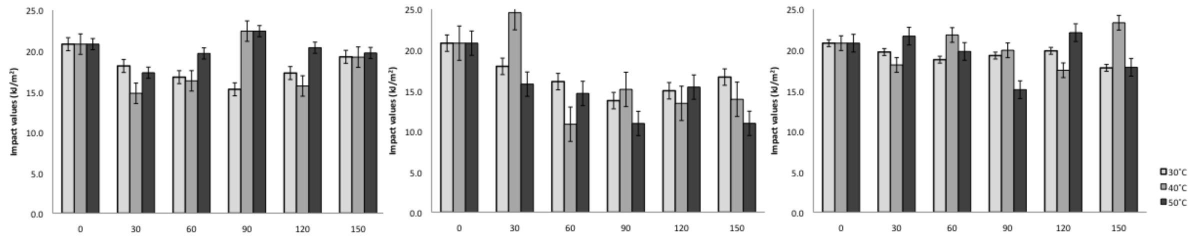

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Figure 2. Impact values (kJ/m^2) of degraded LDPE samples at different ultrasonic temperatures and baths

Effect of ultrasonic temperatures and baths substances on tensile strength of PP and LDPE

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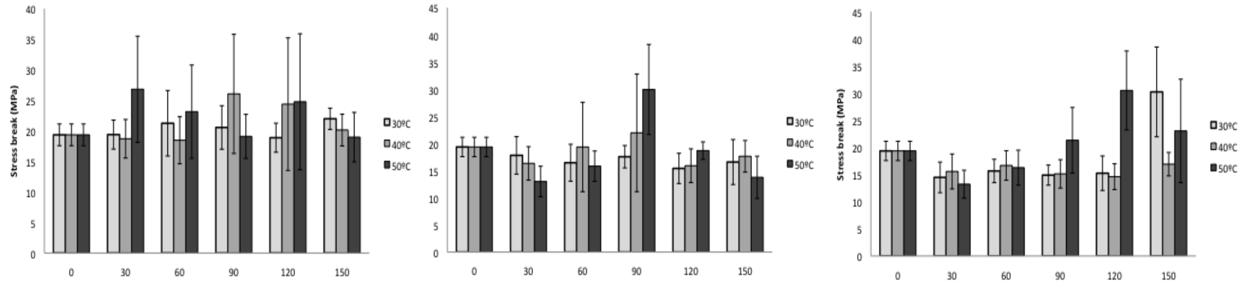

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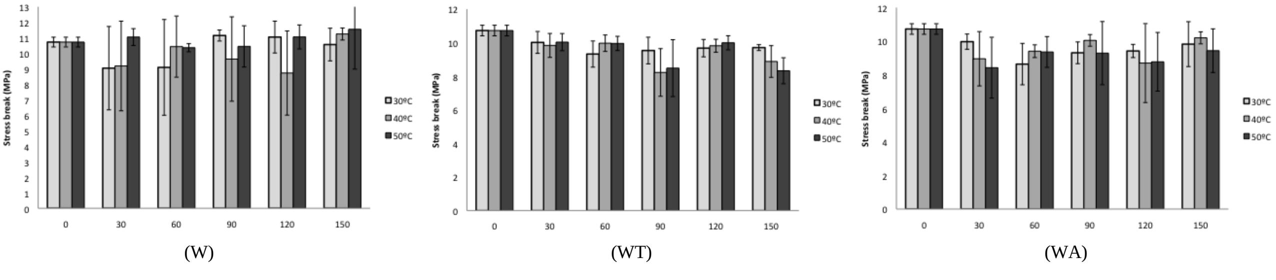
Figure 3. Tensile strength (MPa) of degraded PP samples at different ultrasonic temperatures and baths

Effect of ultrasonic baths on LDPE degraded tensile strength was shown in Figure 4. The LDPE tensile strength in W bath was decreased at 30˚C within 60 min of reaction time. Shorter time was needed to reduce by 16% of pristine samples strength. However, prolong of reaction time shown an increased of tensile strength for all reaction temperatures in W bath. On the other hand, in WT and WA baths, shorter reaction time within 60 – 90 min of reaction will reduce by 22%. Prolong of reaction time shown fluctuated increased of tensile strength. LDPE as non-rigid material responded differently in different bath substances. It was easily broke on applied pulling forces and less breakage residues (white neck) were observed after breakage. Tensile strength shows a higher sensitivity of ultrasonic bath on the degradation behavior of both PP and LDPE.



(W) (WT) (WA)

Figure 4. Tensile strength (MPa) of degraded LDPE samples at different ultrasonic temperatures and baths

Effect of ultrasonic temperatures and baths substances on maximum bending stress and Young Modulus of bending for PP and LDPE

Figure 5 and Figure 6 show the maximum bending stress (MPa) of degraded PP and LDPE samples. The W and WA bath had no significant effect on both PP and LDPE samples within 150 min. On the other hand, WT bath had affected the degradation of PP within 60 min. Temperature and bath substances do not really influenced the maximum bending stress of both PP and LDPE. Both degraded PP and LDPE were able to withstand and distributed the applied load from their crystalline structure. All ultrasonic bath conditions do not change the structural compound of PP and LDPE with the expected crystalline re-arrangement with increment of temperature. Ultrasonic process within 30 - 50˚C is proven to maintain the crystalline arrangement of PP and LDPE.

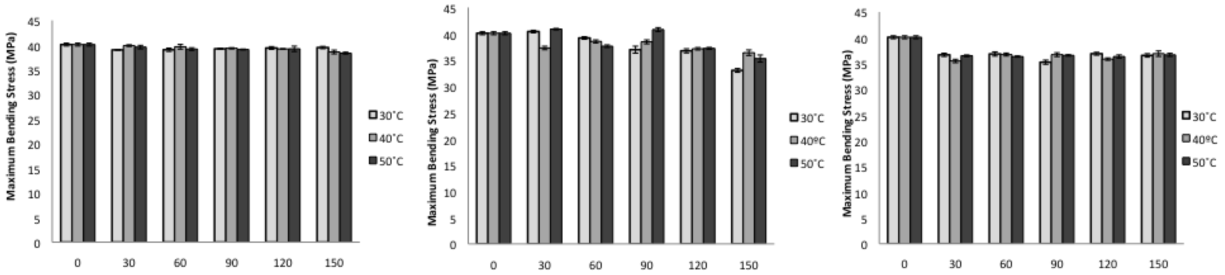


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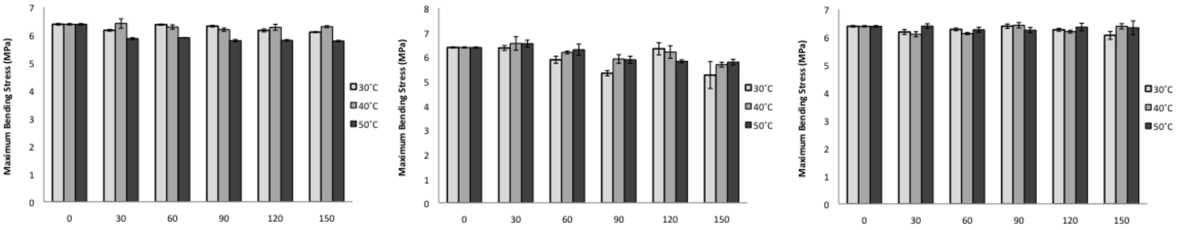
Figure 5. Maximum bending stress (MPa) of degraded PP samples at different ultrasonic temperatures and baths



(W) (WT) (WA)

Figure 6. Maximum bending stress (MPa) of degraded LDPE samples at different ultrasonic temperatures and baths

Figure 7 and Figure 8 show the Young Modulus of bending for both PP and LDPE degraded in different baths. PP with high initial Young Modulus values do not affected by the W bath at early reaction time. Only 6% reduction from pristine PP of 1451 MPa within 150 min reaction for W bath. In WT and WA baths, the highest reduction of 15% from 1451 to 1221 MPa were recorded. WA bath affect the degradation of PP especially at 40˚C while at 30 & 50˚C, gradually decrement and fluctuated values were recorded. Turbulence of bath substances resulted different trend of at the lowest temperature resulted fluctuated degradation. On the other hand, LDPE in W bath had shown gradually decrement prolong of reaction time. In WT and WA similar trend can be observed but at higher temperature of 40 - 50˚C, at 120 min for 4%. Stiffness of PP was not affected by the ultrasonic bath within low temperature range of 30 - 50˚C for 150 min. Hydro-mechanical effect from ultrasonic process doest not accelerate degradation of PP in W bath but minimal effect in WT and WA. On the other hand, LDPE was affected with WT bath resulted in fluctuated values as compared to in W bath. LDPE was more prone to hydro-mechanical effect from ultrasonic process.

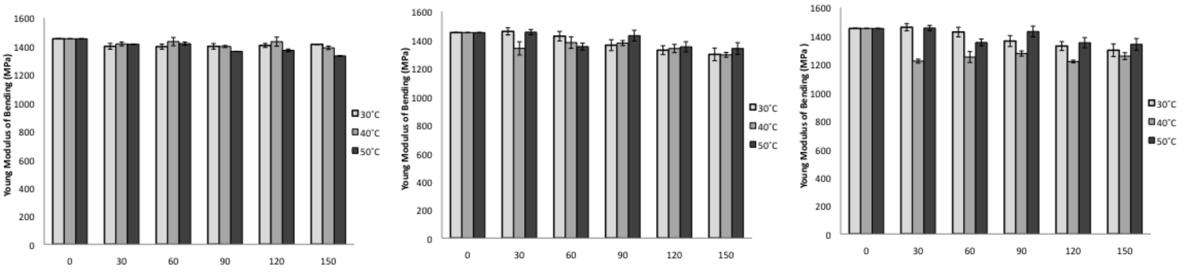


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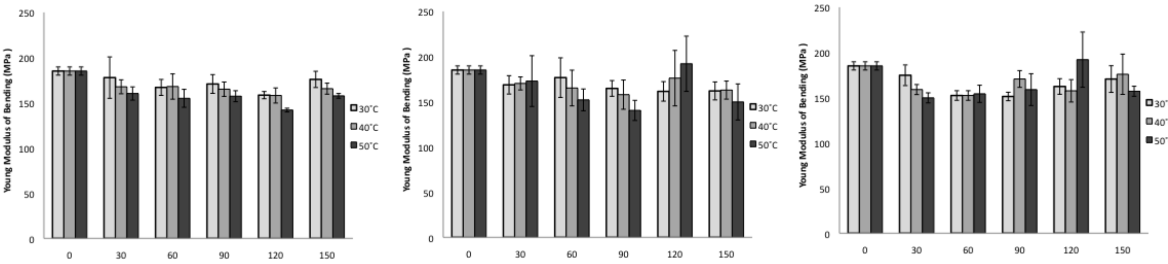
Figure 7. Young Modulus of bending (MPa) of degraded PP samples at different ultrasonic temperatures and baths.



(W) (WT) (WA)

Figure 8. Young Modulus of bending (MPa) of degraded LDPE samples at different ultrasonic temperatures and baths

These mechanical properties of PP and LDPE in all ultrasonic baths were caused by the degradation and rearrangement of molecules responded and influenced by different ultrasonic baths within stipulated reaction time and temperature. This low temperature ultrasonic process had amended the mechanical properties in corresponded to the possible changes of molecular weight without changes in chemical nature of materials. Fluctuated mechanical properties resulted from ultrasonic irradiation that influences the formation, subsequent growth and violent collapse of bubbles or cavities in ultrasonic baths. Generation of free radical from the acoustic cavitations enhanced the process with liquid turbulence associated with liquid shear and affects the degradation. Both hydrodynamic forces and mechanical phenomena of degradation-adsorbed molecules influenced the overall PP and LDPE degradation. On the other hand, increment of tensile strength resulted from stabilization process of polymer chain and ability of molecular rearrangement. Uniform and higher crystalline morphology contributed to stronger and stiffer mechanical properties. Low temperature range ultrasonic degradation resulted in more physical and chemical effects to PP and LDPE.

CONCLUSION

The main findings are LDPE and PP after subjected to ultrasonic degradation is following non-linear pattern in a non-random manner because the semi-crystalline property of the thermoplastics polymer allows partially reversible deformation; the factor of effectiveness of ultrasonic degradation according to time factor is the higher the time, the higher the risk of polymer degradation; while concerning temperature, free radicals are generated more in lower ultrasonic temperature. LDPE and PP responded quite differently to ultrasonic degradation as LDPE is a many-branch PE, while PP has methyl group attached to every other carbon atom on its polymer chain making it stiffer physically but more susceptible to radical attacks. The mechanical effects on the polymers after the process of ultrasonic degradation is non-random and also is confirmed based on the results of mechanical tests; that the ultrasonic treatment parameters can instrument specific change of properties whether it is enhancement or degradation. This is also consistent with a claim from previous study in which ultrasound can be applied for controlled degradation to create or alter polymers with specific properties. Different bath substance clearly shown different degradation trend particularly with the present of toluene and aluminum strap. Ultrasonic degradation is potentially practical to equip both PP and LDPE recycling efforts assisting issues in waste management.

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