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Dynamic Mechanical Analysis of Waste Polyethylene Terephthalate Bottle

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Abstract: Polyethylene terephthalate (PET) led to the disposing and managing issue due to it extensively consumptions. Hence, its application in construction field particular on concrete production has become general. However, the preliminary study on PET itself is very limited. This study was focused on the investigation on mechanical properties of PET film using dynamic mechanical analysis (DMA) over a temperature range of 40-180°C at fixed frequency of 1 Hz. In the paper, glass transition temperature were analyzed with the curves of storage modulus (E'), loss modulus (E") and tan delta. The results show decreasing curve of storage modulus, meanwhile loss storage and tan delta present same curve which is increases with temperature as glassy phase and radically decreases due to rubbery phase. From these curves, the glass transition temperature (Tg) of PET have been characterized which the value has been found to be 114°C. Generally, this study could provide better characterization of a material in order to examine the ability of a material to store or lose energy.
Keywords: Dynamic mechanical analysis, Polyethylene terephthalate, Mechanical properties, Storage modulus,

1. Introduction

Polyethylene terephthalate (PET) is one of the most important and extensively used plastics in the world as it is usually been used in manufacturing beverage container, food and other liquid containers. PET has attracted attention due to its thermo-stability and transparency. However, its widespread consumption leads to the major environmental, economical, and social issue which caused by the challenge of the disposing and managing its waste [1]. Hence, many considerable researches in reusing PET have been done to overcome this issues [2].

PET is widely recycled as a material, making a large contribution to the recycling targets required for plastic [2]. According to Chavan and Rao [3], PET is a transparent polymer that has good mechanical properties and good dimensional stability under different variable load. PET has common characteristics which includes semi crystalline thermoplastic polyester, non-biodegradable, durable, easily processed and handled, low gas permeability, chemically and thermally stable, wear and tear resistant [4].

Attributable to its mechanical strength and durability, PET is extensively used in variety of construction materials such as fiber and aggregate in concrete production. Previous research revealed that PET possess low thermal conductivity value that applicable to be utilized as thermal insulation material in concrete [5]. In this present study, PET will be used as fine aggregate in order to investigate its influences on thermal conductivity of normal strength concrete. A study on mechanical properties of PET is essential to investigate its effect on concrete in details. Hu et al. [6] reported mechanical properties studies related to safety requirement were more fundamental that proper physical properties in building materials improvement.

Dynamic mechanical analysis (DMA) is a direct and effective method to evaluate mechanical properties of polymer materials [6]. Previous research was justified DMA method on semicrystalline PET [7], metallized PET [8], PP/PET blends containing montmorillonite (MMT) [17], annealed and not annealed multilayer film of PET/PE [9], etc. There are few studies on mechanical properties of polymer by using DMA method, however very limited investigations done on PET film and it is fluctuated remarkably [8][9].

To determine the glass transition temperature of PET, an experimental scheme was carried out. Storage modulus (E'), loss modulus (E") and tan delta (δ) varying with temperature were analyzed and will be presented in details in this paper. Finally, important observations and useful values were summarized in the Conclusions.

2. Dynamic Mechanical Analysis

Concerning the determination of mechanical properties of a material, an instrument namely Dynamic Mechanical Analysis (DMA) was created by Poynting in 1909 and it was improved from time to time [19]. DMA, which is commonly acknowledged as thermal analysis technique has been rapidly used to characterize the mechanical properties of any viscoelastic material. Specifically, a variable sinusoidal stress is applied, and the resultant sinusoidal strain is measured [16][19][20]. DMA measures the viscoelastic properties using either transient or dynamic oscillatory test. Viscoelastic behavior normally show phase variances along with amplitudes of stress strain waves used to deduce some material parameters such as storage modulus (E'), loss modulus (E"), complex modulus (E*) and loss tangent (tan δ) [10].

Basically on this stage, mechanical properties such as young's modulus (Y), storage modulus (E'), glass transition temperature (Tg), tensile strength (σ), yield strength (σ_y) can be derived from temperature scan and stress-strain scan at room temperature [6-17]. Varying temperature of the sample or frequency of the stress will cause variation of modulus generally.

In this case, glass transition temperatures were obtained with storage modulus (E'), loss modulus (E") and tan delta (δ) curves. These parameters is proportional to the ratio of energy dissipated to energy stored per cycle of the applied load [14]. The glass transition temperature is characterized by three different ways that is a rapid fall in storage modulus (occurs at lowest temperature which relates to mechanical failure), or a peak in loss modulus (occurs at middle temperature which related to the physical property changes), or a peak in tan delta (occurs at highest temperature). Earlier research found that mechanical properties of polymer materials decrease dramatically above glass transition temperature. This because the effect of frequency which was proportional and more significant than material direction effect [6].

Before starting the DMA experiments, it is important to identify the sample size and shape, static force/ force track, amplitude, frequency and heating rate/ temperature program as experimental considerations. Keeping these into account, the present work has been focused to prepare PET film with appropriate size according to DMA type. The main objective is to study the dynamic mechanical analysis of prepared sample of PET film.

3. Experimental

The experiment of dynamic mechanical analysis is conducted using TA Instruments Q800 DMA. Sample used was obtained from waste PET bottle. The sample in a form of film with 0.17 mm thickness and dimension of 13.16 mm length and 5 mm in width was carried out on DMA deformation modes of tension. In this mode, the sample is placed in tension between a fixed and moveable clamp. Basically, tension clamp is suitable for both films and fibers. The experimental conditions for DMA are summarized in Table 1 which were performed in isothermal condition.

Table 1 Experiment condition for dynamic mechanical analysis of PET film

Parameter	Unit	Range
Temperature range	°C	40 – 180
Heating rate	°C/min	2
Frequency	Hz	1
Strain amplitude	mm	0.01
Force track	%	125
Static Force	N	0.01

The sample was mounted in tension clamp as shown in Fig. 1. After mounted, the furnace was sealed off and directly scanned over a range of room temperature up to 180 °C. The DMA experiment was performed with fixed frequency of 1 Hz and 2 °C min−1 of heating ramp rate for all temperature. Recommend ramp rate for polymer testing is from 1 °C/min to 5 °C/min [16]. The strain amplitude of 0.01 mm within the linear viscoelastic region was imposed to the sample. Force track was used to reduce stretching as sample weakens. Normally, force track values from range 120% to 150% are used for most samples of tension clamp.

All experiment data with input parameters were recorded and processed with equations integrated in a software named PerkinElmer which been set up on personal computer. Storage modulus, loss modulus and mechanical loss factor (tan δ) were logged and plotted against temperature. Originally, to carry out DMA experiment, the procedure was divided into subsequent steps that is fixing specimens, lowering to specified temperature and followed by increasing temperature while acquiring experimental data. The calibrations were implemented according to manual instructions.

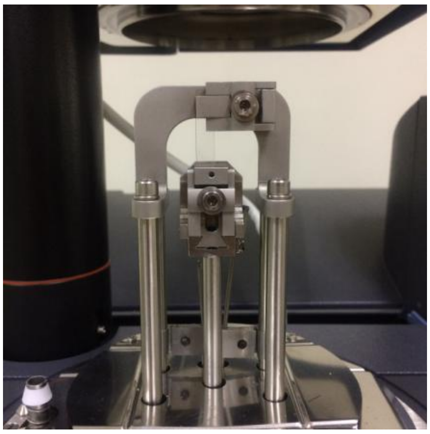

Fig. 1 Sample of PET film in tension clamp fixtures

4. Result and Discussion

The viscoelastic behavior of the PET film was investigated by isothermal DMA experiments within the glass transition range. Fig. 2 present the typical results of DMA scan on PET film as variation of storage modulus, loss modulus and tan delta with temperature. The experiment was performed on temperature ramp method which was analyzed from room temperature, 40°C to 180°C. Observation of these experimental results will be explained in details in the following subsection.

4.1 Storage Modulus

Storage modulus (E') measure the elasticity of material. It is show how the material can store energy. As a function of temperature, storage modulus is an essential and important parameter from DMA in analyzing mechanical properties.

Primary analysis from the curve of sample as shown in Fig. 3(a) reveal that the E' value decreases with increasing the temperature. The value was decreases from 4300 MPa to 350 MPa as temperature increases to 180°C. This may due to the softening of the PET film. The viscoelastic behavior of PET also responsible for steep decrease of E' values at 60°C - 120°C. In general, E' value ranges of the given frequencies (1 Hz) were 4500 MPa and 250 MPa'

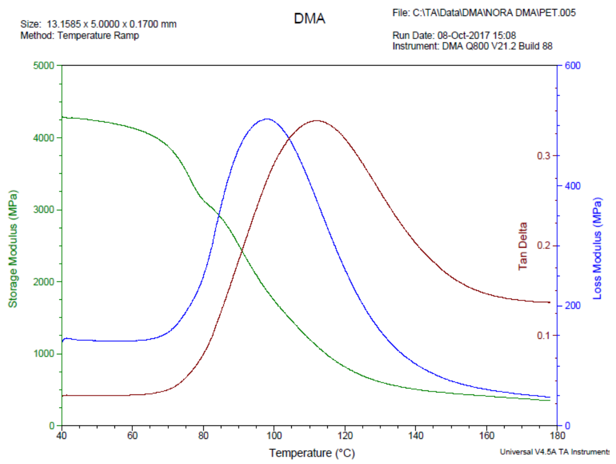

Fig. 2 Typical results of dynamic mechanical experiments of PET film from machine direction.

4.2 Loss Modulus

It is important to understand the idea of loss modulus (E") since glass transition temperature determined with loss modulus was appropriate for molecular analysis [6]. Loss modulus show the ability of the material to dissipate energy which is lost as heat.

As can be seen in Fig. 3(b), variation in E" value show that the loss modulus curves reached a maximum at 500 MPa with the increase in temperature from 60°C to 95°C and thereafter decreased due to the maximum dissipation. This may due to the reflection of molecular processes that show the temperature at the onset of segmental motion.

4.3 Tan Delta

Tan delta was a function of loss modulus and storage modulus. It was used to characterize viscoelastic properties of polymers. Glass transition temperature from tan delta curve was also appropriate for molecular analysis [6]. Glass transition temperature (Tg) is the transition at which material moves from solid to rubbery state. Tg occurs at highest temperature corresponding to peak of Tan δ. On this stage, the height and shape of PET film change systematically with amorphous content.

From Fig. 3(c), it is present that Tg of PET film is 114°C. Obviously, it shows the value of Tan δ initially increases with temperature due to the increase in loss modulus of PET. The energy dissipates as heat once material undergoes softening stage at higher temperature, this led to decreasing of elastic response, which rises the Tan δ up to a maximum value. After that, the glassy phase gradually fluctuations to rubbery phase and led the Tan δ to drastically decreases.

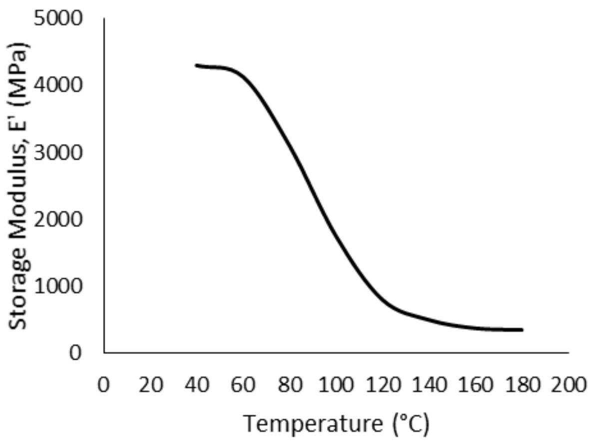

Fig. 3 (a) Storage modulus of PET

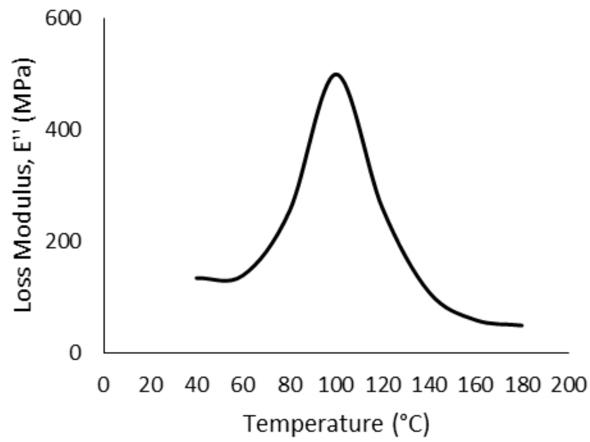

Fig. 3(b) Loss modulus of PET

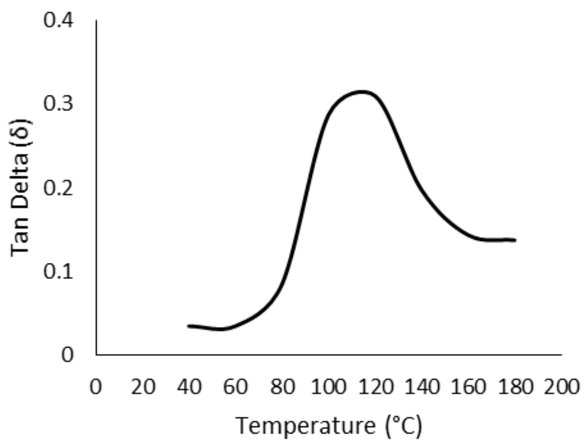

Fig. 3(c) Tan delta of PET

5. Conclusion

This study concerned dynamic mechanical analysis of polyethylene terephthalate (PET) film obtained from waste PET bottle to be employed as fine aggregate replacement in concrete production. Consequently, this study is important to determine the viscoelasticity performance of PET. Hence, the value of storage modulus (E'), loss modulus (E") and tan delta (δ) over a temperature range from room temperature to 180°C at fixed frequency of 1 Hz were collected. These values are useful for understanding the glass transition temperature of viscoelastic material which will be found through the curves. However, some limitations are worth noting. SEM analysis of this plain PET and the reaction of PET with constituent materials of concrete were not developed in this study. Future work will therefore be paid to these aspects to investigate detailed about the chemical and mechanical reactions of the materials combination.

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