



## Study on Moisture Content and Soil Burial Degradation of Waste Polypropylene / Date Seed Particulate (DSP) Composites: Effect of Particle Size and Alkaline Treatment

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### ABSTRACT

Date seed particles (DSP) filled polypropylene composite were prepared by compounding and pressing technique. The average particle sizes of date seeds particles used were 63, 125, 250, 500 and 750  $\mu\text{m}$ . The effects of filler particle size and alkaline treatment on moisture content (%) and soil burial degradation properties of Date seed particles (DSP) filled polypropylene composites were investigated. The result shows that moisture content (%) increases with increase in filler particle size and decreased when sodium hydroxide concentration increases. Soil burial degradation (%) of the composites was studied using percentage weight loss. From the results this property was found to be increasing with the increase in filler particle size and decreased with increase in sodium hydroxide concentration.

**Keywords:** Polypropylene, Date seed particles (DSP), Particle size, Sodium hydroxide treatment, Moisture content and Soil burial degradation properties

### INTRODUCTION

A composite is combination of two materials in which one of the materials, called the reinforcing phase, is in the form of fibers, sheets, or particles, and is embedded in the other materials called the matrix phase. The reinforcing material and the matrix material can be metal, ceramic, or polymer. Composites typically have a fiber or particle phase that is stiffer and stronger than the continuous matrix phase and serve as the principal load carrying members. The matrix acts as a load transfer medium between fibers, and in less ideal cases where the loads are complex, the matrix may even have to bear loads transverse to the fiber axis. The matrix is more ductile than the fibers and thus acts as a source of composite toughness. The matrix also serves to protect the fibers from environmental damage before, during and after composite processing. When designed properly, the new combined material exhibits better strength than would each individual material. Composites are used not only for their structural properties, but also for electrical, thermal, tribological, and environmental applications [1]. Incorporating cost-effective filler in a polymeric matrix will only be feasible if it does not drastically alter the matrix main characteristics such as mechanical properties. The determinant issue here is the compatibility between the interfaces of a biofiller and the polymer matrix. This is important in order to have property-stable composite system. For this reason, various compatibilizers or coupling agents were suggested by some studies in the past to formulate efficient biocomposites based on polymer matrices [2] have shown that incorporating DPML as a coupling agent for the composites of wheat straw / polyethylene (PE) increased its modulus significantly. Wood fiber / polypropylene composites with good mechanical properties may be obtained by using modified coupling agent containing isocyanate functional group [2]. The main impetus in pursuing the use of natural fiber reinforced polymer composites instead of synthetic fiber reinforced polymer composites is the ecological benefit due to the problem associated with their disposal [3]. Recent



advances in natural fiber development are genetic engineering. The composites science offer significant opportunities for improved materials from renewable resources with enhanced support for global sustainability [4]. Natural fibres are categorised based on their origin which are from plants, animals or mineral sources. However, bio-fibres are purely derived from vegetative sources which are fully biodegradable in nature. The main components of bio- fibres are cellulose, lignin, hemicelluloses, pectin and wax. The beneficial engineering properties of bio-fibres are specific strength, low density, high toughness, and good thermal properties as compared to most of the synthetic polymers, reduced tool wear, respiratory irritation and ease of processing. Bio-fibres find many applications in bio medical, food packaging, geotextile, architecture, composites and automotive transportation and general system. Bio- based fibres shows adequate results in terms of mechanical properties. There is the possibility to make composite along with bio-binders to attain requisite materials properties [5]. Natural fibres (Biofillers) also have some demerit when used as a composite filler/reinforcement such disadvantages are; enormous variability in properties, lack of fibre matrix adhesion, poor moisture resistance, poor fire resistance, lower durability, limited maximum processing temperatures, e.t.c. These problems are being dealt with today by carrying out various modifications & treatments. These have different efficiencies for improving the mechanical properties of fibres, the adhesion between matrix and fibre result in the improvement of various properties of final products [6]. The date (*Phoenix dactylifera* L.) has been an important crop in arid and semiarid regions of the world. It has always played an important part in the economic and social lives of the people of these regions. The fruit of the date palm is well known as a staple food. It is composed of a fleshy pericarp and seed. The chemical composition and nutritional value of date flesh have been reported and few works have been published on date palm seeds. However, these were focused on their chemical composition only and not their thermal and sensorial properties. Pits of date palm (seed) are a waste product of many industries, after technological transformation of the date fruits or their biological transformation. In some date-processing countries, such as Tunisia, date seeds are discarded or used as fodder for domestic farm animals. In Tunisia, the mean annual yield of date fruits is about 100,000 tons. From this, around 1000 tons of date seeds oils would be extracted [7].

## MATERIALS

The matrix polypropylene i.e take away containers were obtained at a place of use such as conferences, workshops, restaurants, wedding and naming ceremonies, religion and political gathering e.t.c. as a waste. The filler (date seeds) used in this study were collected from Zaria city market at y'an dabino. Kaduna State, Nigeria.

## METHODS

The matrix (polypropylene) collected were cleaned to remove impurities such as soil and dirt from the containers, air dried and then cut to smaller pieces (flakes form) and kept for future work. Date seeds were cleaned to remove impurities such as soil and dirt, sundried and then ground to particles using jaw crusher and ball mill machines. The date seed particles was then sieved using Impact Lab. Seive (ISO 3310-1:2000, bs 410-1:2000) to



obtain particles sizes of 63, 125, 250, 500 and 750  $\mu\text{m}$ . It was finally dried in hot air oven at 70 °C for 24hours. The fabrication of the composites was carried out by compounding and pressing techniques using two roll mill and compression moulding machines. A mould of (150 × 120 × 5 mm) dimension made from metal was used. The matrix / filler weight fraction was depicted in table 2. The matrix and filler were mixed for 5 minutes in a two roll mill according to the ratio of Table 2 for producing a homogenous composite. The mixture was placed in a metal mould in which mould releasing agent was applied. The press cycle consist of three phases, i.e first phase involved the manual pressing to reduce the mat height, second phase involved in shifting it to the compression moulding machines and finally for cold pressing to facilitate the setting of thermoplastic resin. The maximum pressing temperature, pressure, time and cold pressing or pressure holding time were 160 °C, 5 N/mm<sup>2</sup>, 15minutes and 5minutes respectively [8]. After cold pressing, the resultant composites were removed from the mould for further cooling. The composites panels were then trimmed and put into a hot air oven for conditioning before testing for 48 hours.

**Table 1: Process Parameters and Their Levels**

S/N	Parameters	Unit	Designation		Levels				
			Natural Form	Code Form	A	B	C	D	E
1	Particle size	$\mu\text{m}$	PS	Z <sub>1</sub>	63	125	250	500	750
2	NaOH	%	AL	Z <sub>2</sub>	1	3	5	7	10
3	Flame Retardant	%	FR	Z <sub>3</sub>	5-10	-	-	-	5-10

**Table 2: Experimental Layout Plan**

S/N	Experimental	Code		Process	Parameter	Settings
	Z <sub>1</sub>	Z <sub>2</sub>	Z <sub>3</sub>	PS ( $\mu\text{m}$ )	AL (%)	FL (%)
1	C <sub>1</sub>	C <sub>1</sub>	C <sub>1</sub>	63	-	20
2	C <sub>1</sub>	C <sub>1</sub>	C <sub>1</sub>	125	-	20
3	C <sub>1</sub>	C <sub>1</sub>	C <sub>1</sub>	250	-	20
4	C <sub>1</sub>	C <sub>1</sub>	C <sub>1</sub>	500	-	20
5	C <sub>1</sub>	C <sub>1</sub>	C <sub>1</sub>	750	-	20
6	A	A	A	63	1	20
7	A	B	A	63	3	20
8	A	C	A	63	5	20
9	A	D	A	63	7	20
10	A	E	A	63	10	20
11	B	A	A	125	1	20
12	B	B	A	125	3	20
13	B	C	A	125	5	20
14	B	D	A	125	7	20
15	B	E	A	125	10	20



16	C	A	A	250	1	20
17	C	B	A	250	3	20
18	C	C	A	250	5	20
19	C	D	A	250	7	20
20	C	E	A	250	10	20
21	D	A	A	500	1	20
22	D	B	A	500	3	20
23	D	C	A	500	5	20
24	D	D	A	500	7	20
25	D	E	A	500	10	20
26	E	A	A	750	1	20
27	E	B	A	750	3	20
28	E	C	A	750	5	20
29	E	D	A	750	7	20
30	E	E	A	750	10	20

### Moisture Content (%)

Moisture content of the samples was measured in the laboratory by oven dry method (ASTM D5229). Composites with moisture were placed in an oven Cole Parmer (60648) for 6 hours at 105 °C. Samples were collected every 60 minutes until constant weight was obtained. Moisture content of the samples were calculated using the following equation

$$MC (\%) = \frac{M_{int} - M_{od}}{M_{od}} \times 100 \text{ ----- Scheme 2.1}$$

Where, mc is the moisture content, mint is mass with moisture (g) and mod is mass after drying.

### Soil Burial Test

Soil burial degradation test was carried out in accordance with ASTM D638 standard at departmental research ground demarcated besides spinning work shop between the month of August 2018 and February 2019, by burying the specimens (each 100 x 30 x 5 mm) at a soil depth of about 15cm from the surface ground, and about 30 cm in between each other after taking their initial weight. The specimens were unburied at the interval of seven days unearthen and carefully washed under tap then rinsed with distilled water, dried with soft tissue, oven dried at 70 °C for 1 hour and weight. The above procedure was repeated for twenty four weeks. The percentage weight loss was determined by comparing initial and final weight of the specimens. Soil burial degradation test of the samples were calculated using the following equation

$$\text{Percentage weight loss } (\%) = \frac{W_1 - W_2}{W_2} \times 100 \text{ ----- Scheme 2.2}$$

Where  $w_1$  is the weight before burial and  $w_2$  is the weight after burial

## RESULTS AND DISCUSSION

The results obtained during this research work on the effect of particle size (63, 125, 250, 500 and 750 μm) and alkaline treatment of date seed particles (DSP) at various concentration of sodium hydroxide (1, 3, 5, 7, and 10 %) on Moisture content and Soil burial degradation of the composites are presented



### Moisture Content (%)

The effect of filler particle size ( $\mu\text{m}$ ) and sodium hydroxide (NaOH) treatment (%) on moisture content of the date seed particles / waste polypropylene composites was illustrated in figure 1 and 2. The results shows that composites fabricated with larger particles size filler contained more moisture than those with lower particles as can be seen in figure 1. This property may be due to the poor interfacial adhesion between the filler and the matrix which caused more pores and trapped air during compounding. Composites with lower particles size filler exhibit less content of moisture as a results of larger surface area leading to the better filler / matrix bonding and less particles at the composites surface to attract moisture. Trapped air (voids) and micro-cracks paves a way for moisture to be absorbed into the composites internal structure and these challenges are observed more when larger particles are used. The gaps and flaws at the interfaces, and the micro-cracks in the matrix formed during the manufacturing process boosted up the moisture content as reported by [8]. Few reports were made on the effect of filler particle size on moisture content of composites such as that of [9] who studied the intensity of changes in the moisture content of wood chips in the production of wood polymer composites during drying and storage processes. They reported that the dynamics of drying and moisture absorption is affected by both the type of wood of which the chips are made, and the size of their particles. This probably results from the small size of the chips. These are very small, dusty particles and thus they form themselves in the form of compact layer, to which the moisture is difficult to access. Such a state may be the reason for accomplishing lower moisture content of the wood flour compared to chips. This agreed with [10]. The same increasing pattern of moisture content (MC) was also reported by [11].

The effect of sodium hydroxide (NaOH) treatment on moisture content of date seed particles / waste polypropylene composites was shown in figure 2. The results revealed that sodium hydroxide (NaOH) treatment rendered the composites more resistant to moisture and lower the drying time. Composites fabricated with filler treated with higher sodium hydroxide concentration show less moisture content when compared with those produced with filler treated with lower sodium hydroxide concentration, that is to say moisture content decrease with increasing sodium hydroxide concentration. The higher the concentration of sodium hydroxide from 0 to 10 % the lower the moisture content of the composites. 0 % concentration shows least moisture content compared with 10 % concentration which shows highest moisture content. This decrease in moisture content was as a result of the removal of most of the free hydroxyl group (OH) present in the filler cellulosic structure by sodium hydroxide at higher concentration. This treatment reduced hydrophilic nature of the filler, make it more hydrophobic and thus reduce its percentage (%) moisture content. Similar event was reported by [17]. The effect of alkali treatment on the moisture absorption tendency of single abaca fiber was investigated by Punyamurthy *et al* (2012). The result shown that the alkali treated fiber absorbs less moisture than the untreated raw fiber [18].

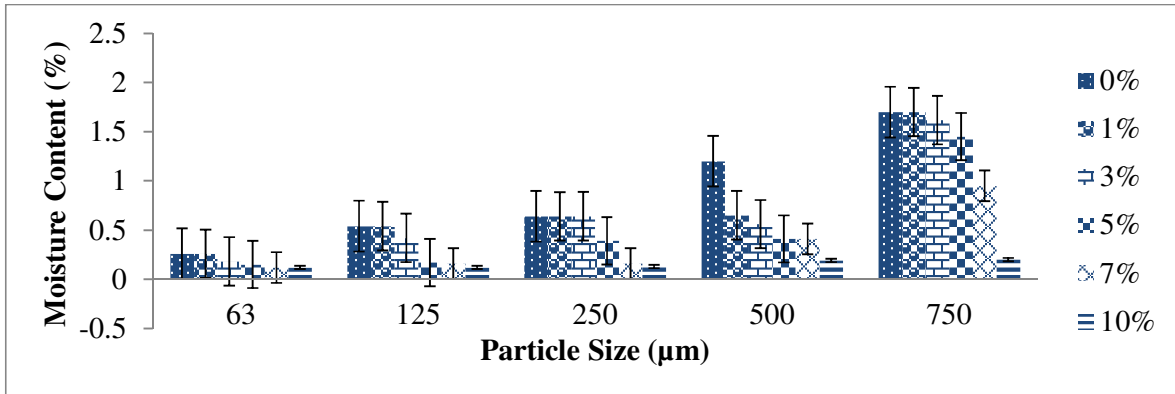


Figure 1: Effect of Particle Size on Moisture Content of PP / DSP Composites

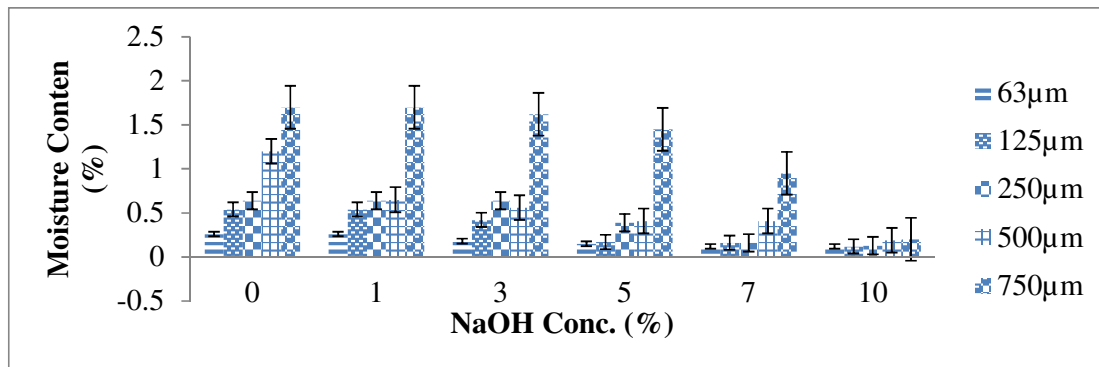


Figure 2: Effect of NaOH Treatment on Moisture Content of PP / DSP Composites

### Soil Burial Test

The results obtained during this research work on soil burial degradation test of the date seed powder / waste polypropylene composites were shown in figure 3 to 13. From the result obtained there was no weight loss during the first, second and third weeks after burial in all the samples. Small decreased in weight of some of the composites were observed after fourth week 0.078, 0.084 and 0.096 % for 250, 500 and 750 µm respectively. Composites produced with larger particle size filler shows rapid degradation than those with smaller particle size filler. Natural fillers open up ways for microbes to act on the composites due to their inherent biodegradability. Pure polymers didn't lose their weight due to non-biodegradable characteristics. Microorganisms mostly consume the fiber parts. The extent of degradation depends on the nature of reinforcing filler, particle size and their modification. These results agreed with the findings of [12]. Composites fabricated with larger particle size filler have more particles at the surface that makes them more vulnerable to microorganism's attack than those fabricated with lower particle size filler. Particle size plays a great role on composites properties, the smaller the particles the larger surface area created and the better dispersion of particles leading to production of more compact material that exhibit better properties. Composites with larger particles are less compact and thus absorbed more moisture that favoured biodegradation process compare with those of lower particles. Additionally, the gaps and flaws at the interfaces, and the micro-cracks in the matrix formed during the manufacturing process by larger



particles size boosted up the moisture content which in turn speeds up the degradation process. Similar event was reported by [13] and [14]. The effect of sodium hydroxide (NaOH) treatment on soil burial degradation of date seed particles / waste polypropylene composites was depicted in figure 9 to 13. The results show that sodium hydroxide treatment on date seed particles impacted resistance to biodegradation on the composites. Composites fabricated with treated filler shows slow degradation when compared with the untreated ones. Decrease in percentage (%) weight loss was observed as the concentration of sodium hydroxide was going up from 0, 1, 3, 5, 7 to 10 %. Maximum percentage (%) weight loss was obtained at 0% concentration to be 0.811 % and the minimum was 0.100 % at 10% sodium hydroxide concentration. Water and temperature are major factors affecting bio-degradation process, absence of water in the composites due to sodium hydroxide treatment slow down the degradation process of the composites. Since most of the hydrogen bondings within the filler cellulosic structure are disrupted during the treatment removing many of the hydroxyl groups thus, minimum water was absorbed by the composites. This makes it very difficult for microorganisms to survive reducing the percentage weight loss of the composites. According to [13] all composites show an increased degradation rate during the buried process. This is likely a result of the penetration of moisture and rain water into the composites, causing the hydrolysis of surfaces and interfaces. The result suggests that the degradability of composites is slightly higher than that of neat PLA. Treated empty fruit bunch fiber-reinforced composite has been found to be more stable than untreated fiber-reinforced composite. [15] investigate the Biodegradation behaviour of natural rubber composites reinforced with natural resource fillers (peanut shell powder and coconut shell powder) monitoring by soil burial test and reported that chemically modified fillers are more resistant to biodegradation since the % of weight loss decreases with chemical treatment, which can be attributed to the non/less availability of hydrophilic centers in the fillers. It can be understood that chemically modified fillers are more resistant to biodegradation as weight loss decreases with chemical treatment. This result is in line with the report made by [16].

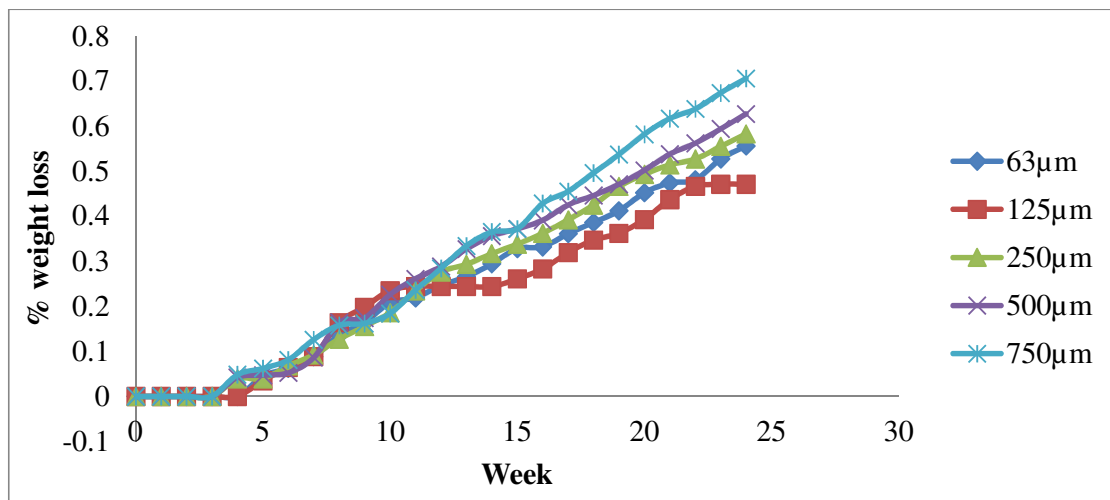


Figure 3: Effect of Particle Size on Soil Burial DEGRADATION of PP/untreated DSP Composites

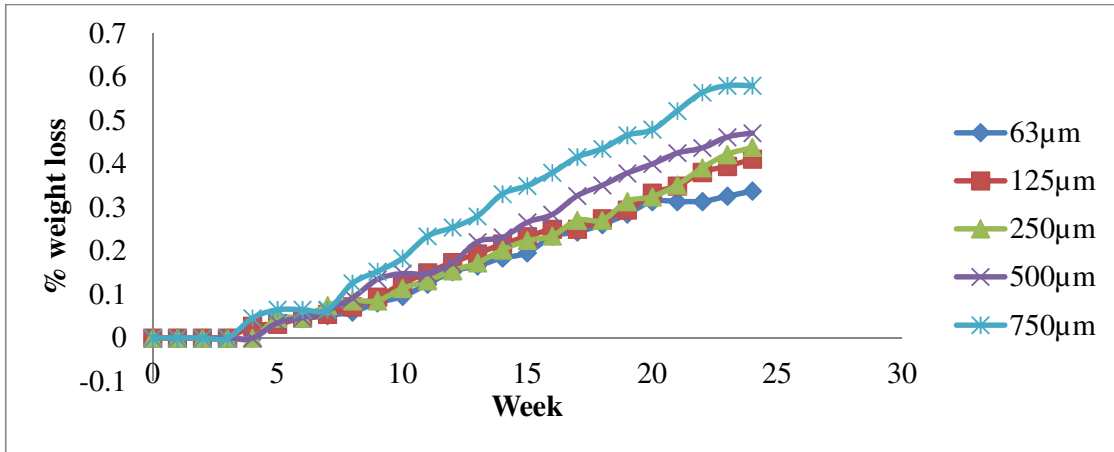


Figure 4: Effect of Particle Size  $\mu\text{m}$  on Soil Burial Degradation of PP / 1% DSP Composites

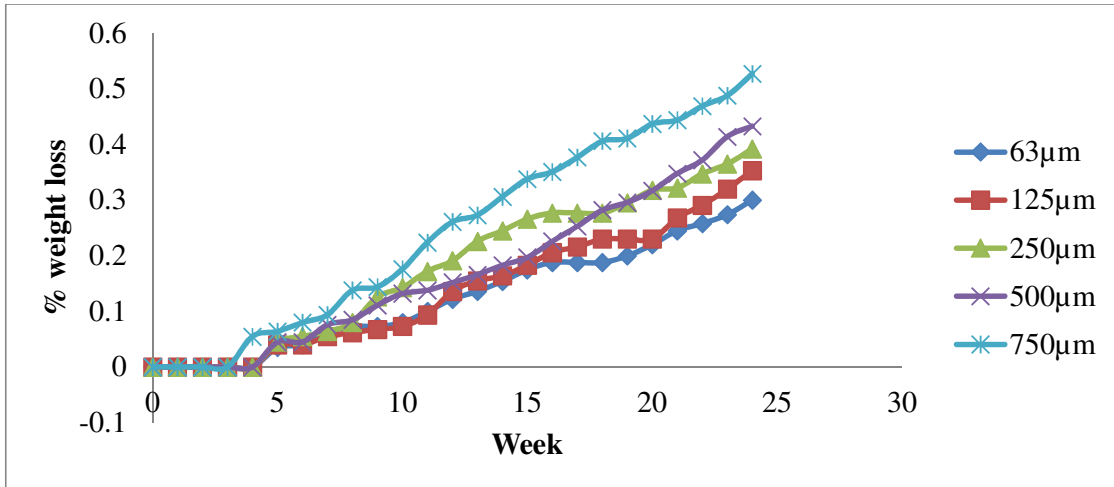


Figure 5: Effect of Particle Size  $\mu\text{m}$  on Soil burial Degradation of PP / 3 % DSP Composites

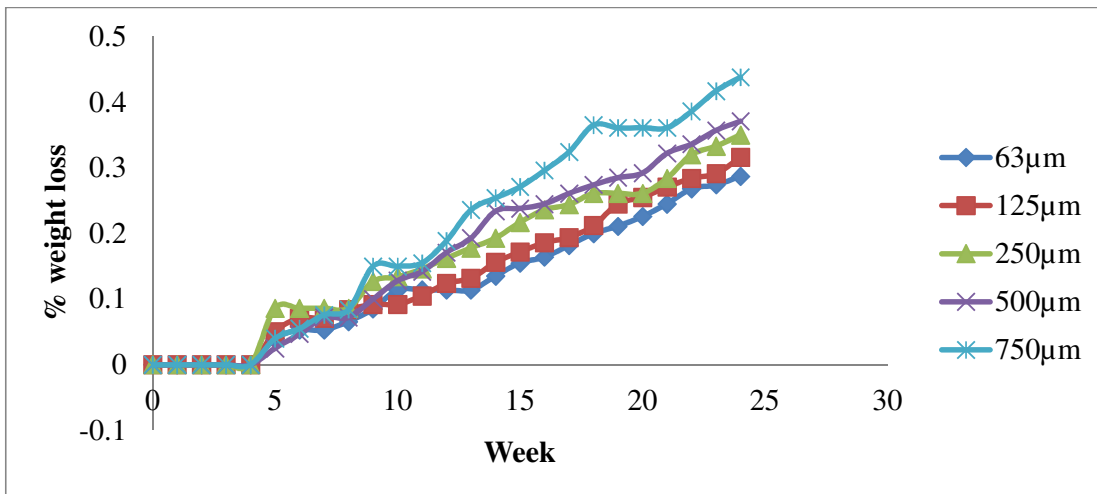


Figure 6: Effect of Particle Size  $\mu\text{m}$  on Soil Burial Degradation of PP / 5 % DSP Composites



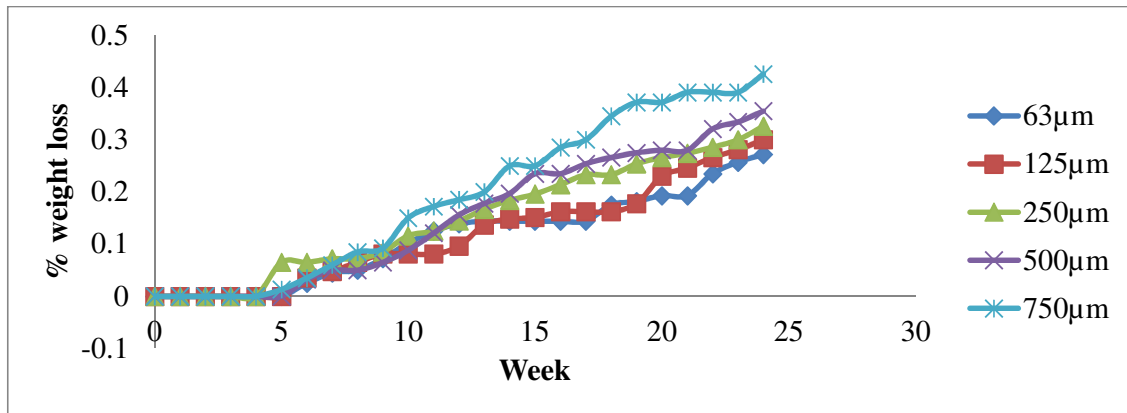


Figure 7: Effect of Particle size  $\mu\text{m}$  on Soil Burial Degradation of PP / 7 % DSP Composites

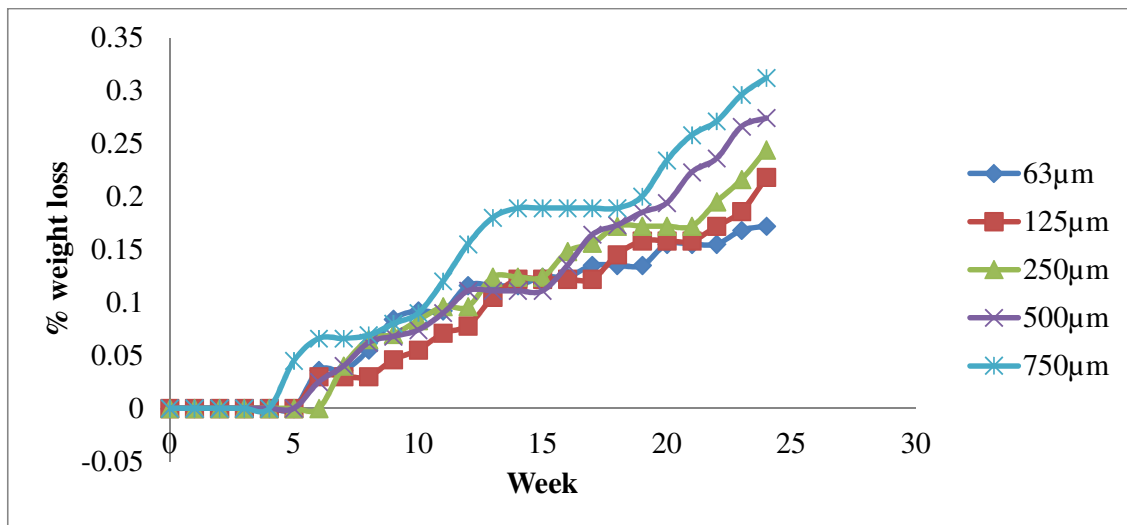


Figure 8: Effect of Particle Size  $\mu\text{m}$  on Soil Burial Degradation of PP / 10 % DSP Composites

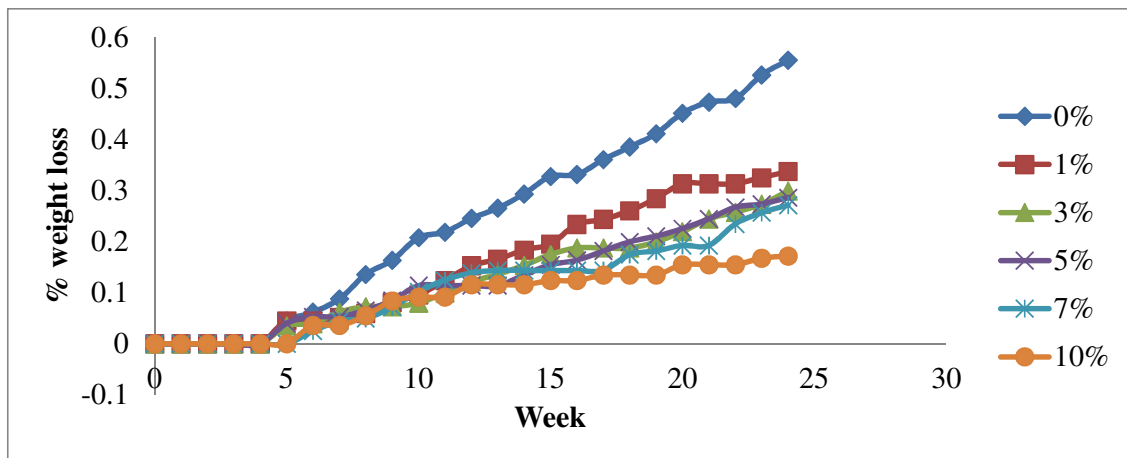


Figure 9: Effect of NaOH Treatment on Soil Burial Degradation of PP / 63  $\mu\text{m}$  DSP Composites

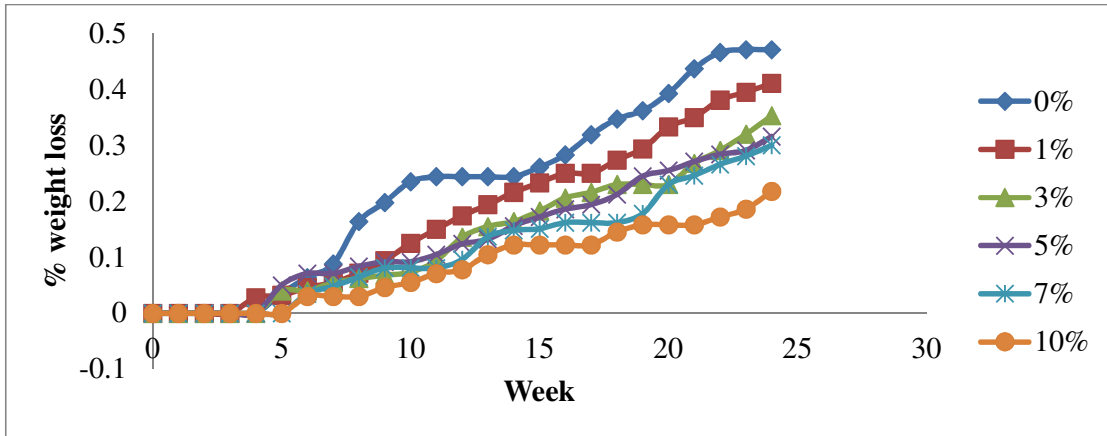


Figure 10: Effect of NaOH Treatment on Soil Burial Degradation of PP / 125  $\mu\text{m}$  DSP Composites

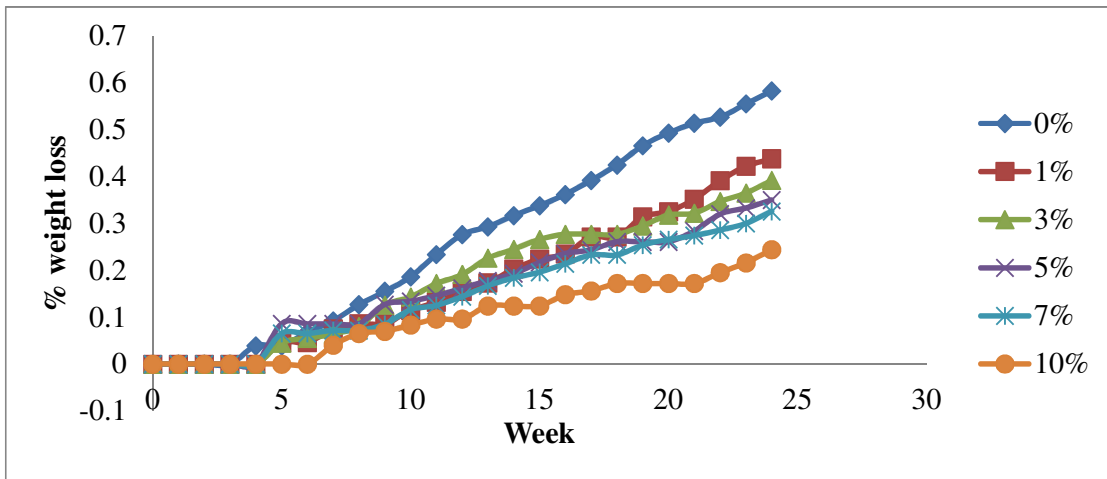


Figure 11: Effect of NaOH Treatment on Soil Burial Degradation of PP / 250  $\mu\text{m}$  DSP Composites

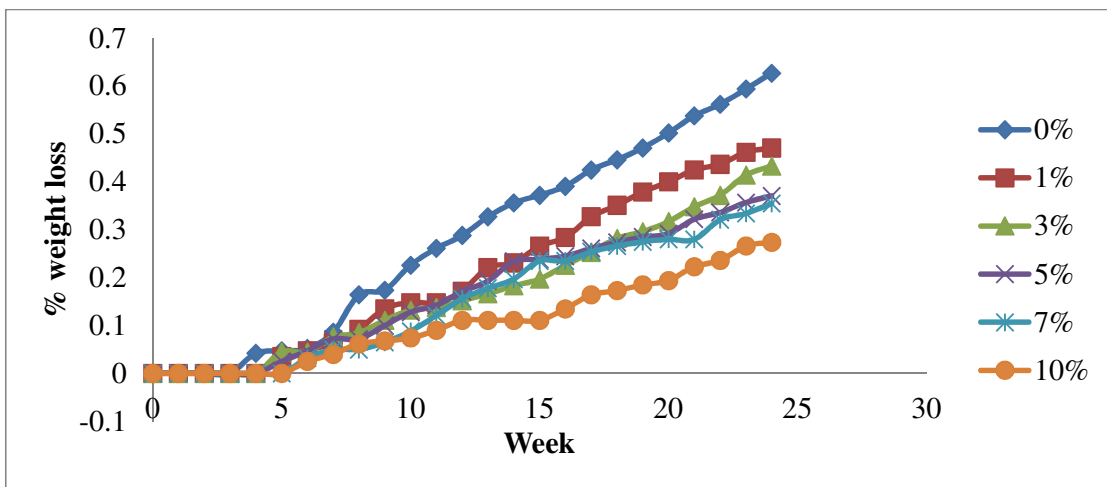


Figure 12: Effect of NaOH Treatment on Soil Burial Degradation of PP / 500  $\mu\text{m}$  DSP Composites

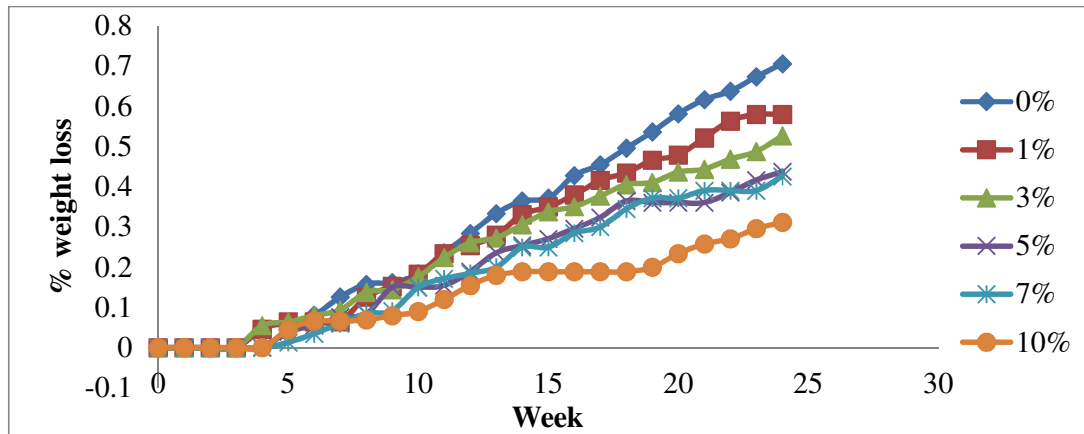


Figure 13: Effect of NaOH Treatment on Soil Burial Degradation of PP / 750  $\mu$ m DSP Composites

## CONCLUSION

Date seed particles (DSP) / waste Polypropylene (wPP) composites were produced by compounding and pressing techniques. The particle size of date seeds used were 63, 125, 250, 500 and 750  $\mu$ m. The effects of filler particle size and alkaline treatment on moisture content (%) and soil burial degradation weight loss (%) properties of Date seed particles (DSP) waste Polypropylene (wPP) composites were investigated. The composites were characterised by oven dry and percentage (%) weight loss methods. The results shows that the moisture content (%) increase with increase in filler particle size and decrease with increasing sodium hydroxide concentration. Bio-degradability properties after six months of soil burial degradation test did not show unacceptable properties and the changes in the weight loss (%) of the tested composites (un-buried and buried) were minimal.

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