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SORPTION RESISTANCE OF *FICUS POLITA* SEED POWDER AND CALCIUM CARBONATE FILLED POLYPROPYLENE

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ABSTRACT

The aim of this research is to determine the influence of Calcium Carbonate and *Ficus polita* powder on the sorption resistance of Polypropylene Composites. The composites were prepared by mix melting and compression molding techniques. The compressed molded articles without and with fillers, PP, (PP/$CaCO_3$) and (PP/FPSP) of different compositions (10/90, 20/80, 30/70, 40/60, 50/50, 60/40, 70/30 and 80/20) were characterized for, water absorption resistance capacity at the different fillers' loading. Comparative studies were made on the sorption strength of the pure polypropylene (PP), polypropylene/calcium carbonate (PP/$CaCO_3$) and polypropylene/*ficus polita* powder (PP/FPSP). The surface sorption characteristics of calcium carbonate and *ficus polita* powder has been investigated and the highest percentage was recorded at 20/80 of PP/FPSP (90%). There was gradual increase of the percentage sorption of water from unfilled polymer matrix to polymer composites. It was generally observed that for composition of 90/10, 80/20, 70/30, 60/40, 50/50 and 40/60 of both composites (PP/$CaCO_3$ or PP/FPSP) as the filler's loading, the % sorption of water also increases, with PP/FPSP having the least % sorption resistance (highest % of water absorbed). The sorption data for 20/80 and 30/70 PP/FPSP compositions showed large increase of the percentage of absorption of water. The PP and PP/$CaCO_3$ composites have the best sorption resistance compared the pristine PP and PP/FPSP composites. Thus, it can be established, that, the PP with % sorption of 10 % water and 0.1 % in 0.1 M NaOH and 90 gPP/10 g$CaCO_3$ with %sorption 0.1% in water and 0.1 M NaOH, this mean that the polypropylene composites samples have good % sorption resistance and hence they can be used for industrial application where sorption resistance is applicable except 20/80 PP/FPSP composition (90 % water sorption) that has the least % sorption resistance.

Keywords: Polypropylene, *Ficus-polita*; Calciumcarbonate; composite and Compression Molding Machine

INTRODUCTION

Both technically and economically, fillers form a large and increasingly significant part of the polymer industry, for both plastics and elastomers. There have been wide-ranging developments, covering the chemistry and formulation of new and more efficient fillers systems and the safer use of additives, both by processors in the factory and, in the wider field, as they affect the general public. It has also become clear that, to meet today's requirements, the budgets for research and development and the structure needed to maintain a global presence are beyond the resources of individual companies, resulting in many mergers and takeovers, leading to the creation of a few world-scale giant producers, complemented by a number of specialists. Solvent sorption is one of most widely used techniques in characterizing structural changes in polymers. According to Gray and Gilbert (1975), the rate of solvent absorbed at equilibrium all depend on the type of polymer and its thermal or shear history. Illers (1977) reported that in semi crystalline polymers, solvent sorption behavior is directly proportional to the amount of crystallinity and the available free volume in a polymer. Measurements of the equilibrium sorption of vapor or of gases in miscible blends can, in principle, give information about the interaction energy parameter, the rate of sorption of a liquid by a resin following Fick's law of diffusion (Vergaud, 1991). Many plastics and composites are widely used because of their chemical resistance, low weight and mechanical strength verily, thermoplastics such as polypropylene is widely use in chemical plants, in distribution systems for natural gas and water, and in effluent and sewage disposal networks. The chemical interactions with plastics materials can be categorized into direct chemical attack, selective chemical damage, environmental stress cracking (ESC), surface detract and swelling (Birley, *et al.*,1992). Combined effects of stress cracking and photodegradation in polystyrene was recently reported by (Sousa *et al.,* 2005) and they found out that butanol causes significant modification in PS with extensive surface cracking as well as reduction in mechanical properties. Studies on chemical resistance of plastics composites including PP/$CaCO_3$ composites have received very little attention among the researchers. Therefore, the present study was carried out to investigate the effects of $CaCO_3$ and FPSP loadings on the chemical resistance of PP. Two chemical reagents from different groups were used in this study, water which is a neutral and universal solvent and sodium hydroxide from alkaline solution.

There have been many studies using nano calcium carbonate to enhance the properties of polymers. Among the techniques employed to disperse the nano filler include in-situ polymerisation, melt mixing using internal mixers and melt compounding using twin-screw extruders. In-situ polymerisation technique has been used for PVC and PET, while melt mixing and melt compounding appeared to be the preferred method for polypropylene (Ritchie, 1993).

Eiras and Pessan (2009) presented a paper on the effects of calcium carbonate nano particles in the crystallization of polypropylene. The experimental work includes Differential Scanning Calorimetry analysis of isothermal and non-isothermal crystallization, optical microscopy and X-ray diffraction. In their study, four compositions of PP/$CaCO_3$ nano-composites with calcium carbonate content of 3%, 5%, 7%, and 10% by weight were prepared in a co-rotational twin screw extruder machine (Weiner and Pfleirer ZSK-30) with temperature profile of 170/190/190/190/190/1950 C, and a screw speed of 100 rpm. DSC analysis were conducted by heating the samples from 300 ºC to 2000 ºC at a heating rate of 100 C/min keeping the sample at this temperature for 2 minutes and then cooling down from 2000 ºC to 300 ºC at a cooling rate of 100 C/min. The isothermal analysis and optical microscopy analysis were conducted by heating the sample at the same condition and then cooling from 2000ºC to their crystallization temperature, maintaining it at this temperature for 15min. The ductile deformation of the matrix is inhibited because of the presence of CRH. Three different mechanisms have been proposed for moisture penetration into the composite and the main process is the diffusion of water molecules into the composite, while the main process is the diffusion of water molecules inside the micro gaps between the polymer chains. The other two mechanisms are capillary transport of water into the gaps and flaws created at the interface of fibre and polymer matrix, because of incomplete wettability and impregnation and also diffusion of water molecules into the microcracks formed in the matrix during the compounding process (Liang, 2002).

Experimental Procedure

The sorption test was conducted according to ASTM D 1037-99. Water absorbed was determined by the immersion of 1g of 100 × 12 ×4 mm dimensions of the compressed molded samples in to specimen bottles containing distilled water 0.1 M sodium hydroxide solution at 25 ± 2 ℃ temperature for 72 hours. The weight increase was periodically measured. The amount of water and sodium hydroxide absorbed by the composites at the specified time and temperature was calculated according to the following equation:

$$M_t(\%) = \frac{W_t - W_0}{W_0} \times 100 \qquad (1)$$

Where M_t is the amount in percentage of water and sodium hydroxide solution absorbed at time t, w_t is the weight of the specimen at time t and w_0 is the initial weight of the specimen at time t_o.

Table of formulation

Table 1

S/No	Mass of Ingredients (g)	1	2	3	4	5	6	7	8	9
1	Polypropylene	100	90	80	70	60	50	40	30	20
2	*Ficus polita* seed powder	0	10	20	30	40	50	60	70	80

Table 2

S/No	Mass of Ingredients(g)	1	2	3	4	5	6	7	8	9
1	Polypropylene	100	90	80	70	60	50	40	30	20
2	Calcium Carbonate	0	10	20	30	40	50	60	70	80

Results of Experiment

Table 3: Sorption Test Results (PP/FPSP)

S/No	Filler loading(g)	Initial mass of PP/ FPSP, W_o (g)	Final mass of PP/FPSP, W_1 (g)	Mass of water absorbed (g)	% Sorption Efficiency $\frac{W1-Wo}{Wo}$ x 100
1	0	1	1.001	0.001	0.01
2	10	1	1.011	0.011	1.10
3	20	1	1.02	0.020	2.0
4	30	1	1.02	0.02	2.0
5	40	1	1.04	0.04	4.0
6	50	1	1.05	0.05	5.0
7	60	1	1.5	0.50	50
8	70	1	1.6	0.6	60
9	80	1	1.9	0.90	90

Table 4: Sorption resistance Test Results (PP/$CaCO_3$)

S/No	Filler loading(g)	Initial mass of PP: $CaCO_3$, W_o (g)	Final mass of PP/ $CaCO_3$, W_1 (g)	Mass of water absorbed (g)	% Sorption Efficiency $\frac{W1-Wo}{Wo}$ x 100
1	0	1	1.001	0.001	0.1
2	10	1	1.001	0.001	0.1
3	20	1	1.002	0.002	0.2
4	30	1	1.003	0.003	0.3
5	40	1	1.005	0.005	0.5
6	50	1	1.010	0.010	1.0
7	60	1	1.030	0.030	3.0
8	70	1	1.550	0.300	30
9	80	1	1.250	0.550	55

Table 5: Effect of NaOH Solution of Sorption resistance (PP/CaCO$_3$)

S/No	Filler loading(g)	Initial mass of PP/ CaCO$_3$, W$_o$ (g)	Final mass of PP/CaCO$_3$, W$_1$ (g)	Mass of NaOH(aq) absorbed (g)	% Sorption Efficiency $\frac{W1-Wo}{Wo}$ x 100
1	0	1	1.001	0.0010	0.1
2	10	1	1.001	0.0010	0.1
3	20	1	1.001	0.0010	0.1
4	30	1	1.0020	0.0020	0.2
5	40	1	1.0025	0.0250	2.5
6	50	1	1.0100	0.100	10
7	60	1	1.2000	0.2000	20
8	70	1	1.4000	0.4000	40
9	80	1	1.5000	1.5000	50

Table 6: Effect of NaOH Solution Sorption Test Results (PP/FPSP)

S/No	Filler loading(g)	Initial mass of PP/ FPSP, W$_o$ (g)	Final mass of PP/FPSP, W$_1$ (g)	Mass of NaOH(aq) absorbed (g)	% Sorption Efficiency $\frac{W1-Wo}{Wo}$ x 100
1	0	1	1.1	0.10	10
2	10	1	1.11	0.11	11
3	20	1	1.2	0.12	12
4	30	1	1.2	0.20	20
5	40	1	1.24	0.24	24
6	50	1	1.22	0.24	24
7	60	1	1.5	0.50	50
8	70	1	1.6	0.60	60
9	80	1	1.65	0.65	65

Sorption Results

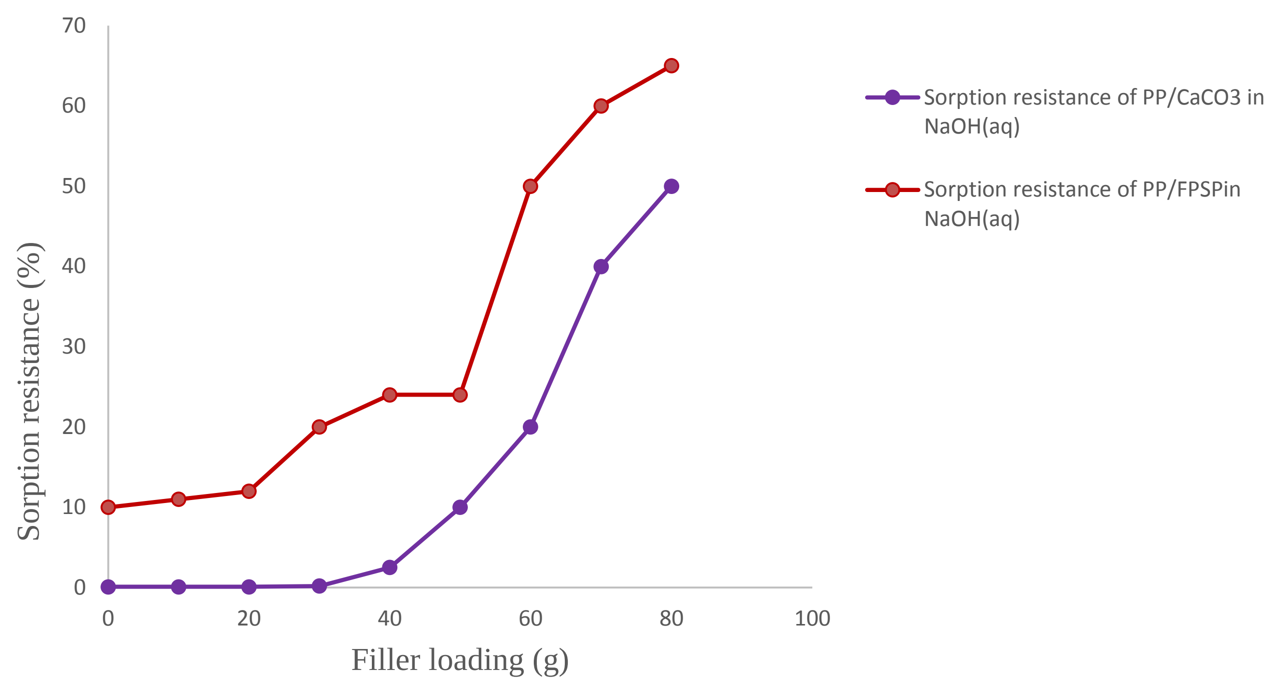

Figure 1: Effect of filler content on the sorption resistance of PP, PP/$CaCO_3$ and PP/FPSP composites in (0.1 M NaOH)

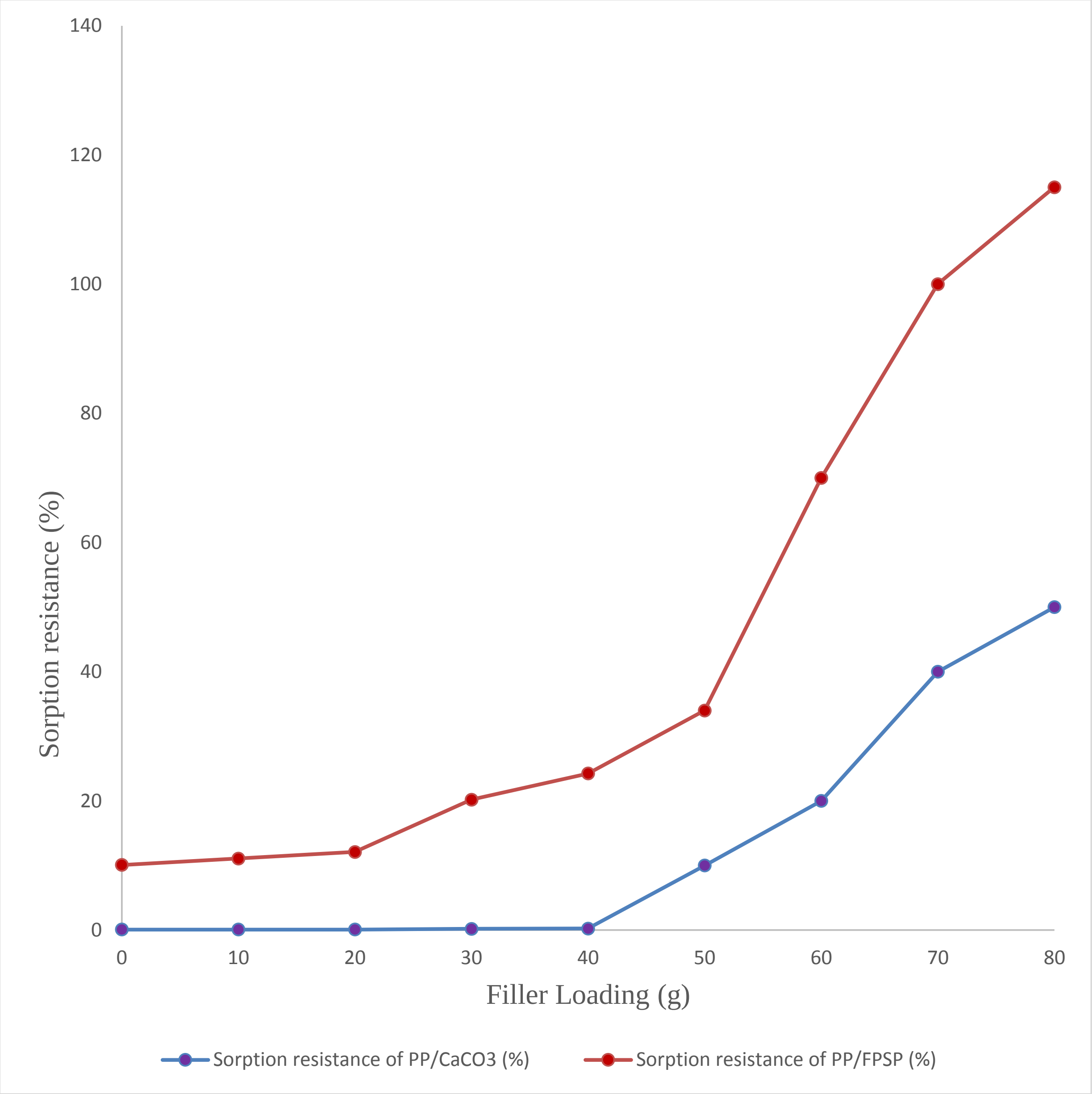

Figure 2: Effect of filler loading on the sorption resistance of PP, PP/$CaCO_3$ and PP/FPSP composites in water

DISCUSSION OF RESULTS

Tables 3 give the sorption data of the unfilled polypropylene (PP) and filled PP/$CaCO_3$ and PP/FPSP composites in water. The % absorption values were plotted against the various compositions in Figure 1 which gives the sorption graph of the sorption test of the polymer matrices. Sorption data in water (H_2O) gave a weight gain, the polymer composites (both unfilled and filled) witnessed a weight gain due to their absorption of water. However, solvent absorption increases with filler content. The explanation for this is that the water molecules slowly diffused into the polymer composites structure leading to high polymer-polymer and polymer – solvent intermolecular forces as a result of strong hydrogen bonding as observed by Ashraf (2007). Three different mechanisms have been proposed for moisture penetration into the composite (two or more physically distinct and mechanically separable materials). The main process is the diffusion of water molecules inside the micrographs between the polymer chains. The other two mechanisms are capillary transport of water into the gaps and flaws created at the interface of fibre and polymer matrix because of incomplete wettability and impregnation and also diffusion of water molecules into the micro-cracks formed in the matrix during the compounding process (Liang, 2002).

There was gradual increase of the percentage sorption of water from unfilled polymer matrix to polymer composites in which we observed that for composition of 90/10, 80/20, 70/30, 60/40, 50/50, 40/60 there was gradual rise of water absorption as the filler ratio increases for PP/FPSP while the sorption data for 30/70 and 20/80 PP/FPSP compositions showed large increase of the percentage of absorption of water. This observation revealed that, interaction has taken place between the polypropylene matrices and the cashew nutshell powder filler which might have altered the molecular order of PP/FPSP composites at these compositions compared to those cases of the same compositions for PP/$CaCO_3$. Although, in the case of PP/$CaCO_3$ the constant values of sorption observed for 80/20, 70/30, 60/40, 50/50 and 40/60 revealed that no interaction has taken place to have altered the molecular order of PP/$CaCO_3$ composites composition compared to the case of PP/FPSP (Table 5 and 6) whereas increase in the weight of the $CaCO_3$ in polypropylene matrix increases the % sorption of water indicating slight change of molecular order at higher filler loading (both $CaCO_3$ and FPSP) even though the increase was not as much as for PP/FPSP compositions.

Similar result has also been reported by Mamza, (2011), he conducted sorption test using four different solvents (i.e dil. HCl, NaOH, CH_3COCH_3 and H_2O) on PS/PVAc blends filled with alpha cellulose (both unfilled and filled), he reported that in all the four solvents, sorption increased, except acetone showed a dramatic decline in % absorption as the filler loading was increased. The equilibrium of absorbed solvents can be attributed to the level of polymer-polymer interactions. In the study, acetone seemed to dissolve the crystalline portion of the blends and could not be used to detect the differences between the polymer blends and their molecular structure, while water, HCl and NaOH which can swell the amorphous regions of the polymer but will not dissolve the blend crystallites, can be used to detect differences between the polymer blends and their molecular structure (Mamza, 2011).

The subsequent rise in sorption values was attributable to the saturation of the gap between polypropylene matrix and the filler which assisted inter-grain friction in the melt, thereby increasing the gelation level. The amount of water absorption of the composites increased with the incorporation of filler in the polymer matrix. The extent of solubilizing the composites is dependent on the nature of solvents used and the filler addition into the polymer composites structure (Figure 1). It is evident from these results that the polymer composites – solvent interactions were more dominant in the presence of the solvents over polymer –filler interaction. It was also therefore established further for this study that sorption parameters of PP/$CaCO_3$ and PP/FPSP composites were largely dependent on filler types and the dependence were in the order, calcium carbonate> cashew nutshell powder. The PP/FPSP had the highest % water absorption for the filled polypropylene composites but a general trend of increase in % water absorption was observed for all polypropylene-filled composites for both or all fillers (i.e. $CaCO_3$ and FPSP) except for PP/$CaCO_3$ that gave a constant % of absorption of water due to the reasons earlier deduced. Similar trend was reported by Jacob *et al.,* (2018) in which the water absorption increases with increasing weight fraction of filler (banana peeled powder) incorporated into recycled High Density polyethylene composites

It can be established here that Sorption can be used to detect the structural changes in the PP/$CaCO_3$ and PP/FPSP composites this is because of the higher adhesive force between PP and $CaCO_3$ as compared to that of PP and FPSP respectively.

Effect of Sodium hydroxide solution (0.1 M NaOH) on Sorption resistance of PP/$CaCO_3$ and PP/FPSP composites

Sorption is a type of chemical quiescent which normally takes place before total dissolution (Samsudin *et al.,* 2005 and Jacob *et al.,* 2018). The sorption effect can be represented by the percentage of weight increase after total submerging of the composites in the appropriate solvent. The effect of the sodium hydroxide solution on the pristine PP after 24 hrs immersion is given in table PP/FPSP slightly swelled up and the swelled up increasing amount of FPSP in the PP matrix compared this trend was observed only at both 20 gPP/80 g$CaCO_3$ and 20 gPP/80 gFPSP which implies that the increase in size was not significant until the filler loading for both of the two fillers used reached 80g each. However, the chemical resistance toward the solution of sodium hydroxide weaken as it was absorbed by the composites with large quantity of the filler particularly for the 20 gPP/80 gFPSP composites composition, this could be attributed to the presence of sodium, Iron, Calcium, Magnesium and phosphorus as detected and reported in the proximate analysis of the *ficus polita* seed powder carried out by (Ndamitso *et al.,* 2010). It was also observed that as the time increases the percentage of sodium hydroxide solution absorbed increases. However, at high PP loading, after immersion in the sodium hydroxide solution is only minimal (less than 1 %) at all the designated experimental periods which depicts that PP is inert to towards sodium hydroxide solution.

CONCLUSION

The present study has generally investigated the effect of the filler content in the polypropylene on the Sorption Resistance capacity and the conclusions were Sorption Resistance capacity of PP/$CaCO_3$ gave higher values compared to PP/FPSP composites. It has also been found that the % sorption of the composites samples were comparable at (10-50 g) filler loads for both $CaCO_3$ and FPSP whereas between (60-70 g) of the two fillers used have water sorption 50% and (80 g) filler loads having 90 % water absorption with respect to *ficus polita* powder filler having the poorest sorption resistance capacity. The best sorption resistances were recorded towards solution of sodium hydroxide for the two fillers' loading regardless of the experimental time except for 20 gPP/$CaCO_3$ and 20 gPP/FPSP composites compositions respectively.

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