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Development of Poly-Lactic-Acid (PLA) Composite Films Filled with Aerosil / Wollastonite on Characterization

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ABSTRACT

This research presents on the two different fillers composites filled with aerosil and wollastonite fibers respectively were dispersed on to the Polylactic acid (PLA). Two different systems were prepared such as system-1(i.e. PLA + aerosil) and system-2 (i.e. PLA + wollastonite). In this connection synthesis of polylactic acid solution and development of composites were prepared with aerosil as filler. Tensile strength and modulus were examined with the help of Instron Universal Testing Machine. Using hand layup technique was used to prepare the composite. Glass moulds are used for casting purpose. Tensile strength and tensile modulus and chemical resistance were measured for the above mentioned two different systems. PLA composite film thick was increased from 1-1.5mm for all the composites. Aerosil and wollastonite filler ratios were considered as 0.1, 0.2, 0.25, and 0.3 wt.% respectively for both the systems. Among two systems system-2 got significantly good performance than the system-1. And also weight of the system-1 is lesser than that of the system-2 as aerosil has high specific volume when compared with wollastonite filler.

Keywords: PLA, Aerosil, Wollastonite, Tensile Strength, Tensile Modulus, Chemical Resistance

I. INTRODUCTION

Polylactide (PLA) is rigid thermoplastic polyester with a semicrystalline or completely amorphous structure depending on the stereopurity of the polymer backbone. PLA has gained a considerable interest due to its bioresorbability, biodegradability, and biocompatibility. Furthermore, its ability to be crystallized under stress, thermally crystallized, filled, and copolymerized, turn it into a polymer with a wide range of applications. PLA is a biodegradable polymer, made from renewable sources, with either a semi-crystalline or amorphous structure. Although these features make PLA an appropriate candidate for food packaging there are, however, some important issues that should be overcome such as poor thermal stability, low mechanical resistance, and limited gas barrier properties. Based on results reported in the literature, the addition of nano-sized fillers (clays) can efficiently improve the mechanical and barrier properties provided that they are well dispersed in the matrix and form an exfoliated structure. On the other hand, this work shows that the incorporation of organically modified clay into PLA enhances the rate of degradation and hence markedly decreases the thermal stability of the resulting nanocomposites. Therefore, control of PLA thermal degradation is another challenge in developing PLA-clay nanocomposites. Packaging materials have been, to a large extent, based on non-renewable materials. The only widely used renewable packaging materials are paper and board which are based on cellulose, the most abundant renewable polymer world-wide. However, major efforts are under way to identify alternative non-food uses of agricultural crops and the production of packaging materials, based on polymer from agricultural sources, could become a major use of such crops. Indeed such alternative bio-based packaging materials have attracted considerable research and development interest for long time and in recent years the materials are reaching the market. Among all bio-based biodegradable polymers studied, poly (lactic acid)

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(PLA) appears to be one of the most attractive polymers commercially available, because of its iodegradability, ease of processing, transparency and price. In general, commercial PLA grades are copolymers of poly (L-lactic acid) and poly(D,L-lactic acid), which are produced from Llactides and D,L-lactides respectively. The ratio of L-enantiomers to D,L-enantiomers is known to affect the properties of PLA, i.e. whether the materials are semicrystalline or amorphous. There is increasing interest in using PLA for disposable degradable plastic articles; however, there are properties such as flexural properties, gas barrier properties, high melt viscosity and melt strength/'elasticity' during processing, that are often not good enough for some end-use applications, such as blow molding. To improve the physical properties of PLA, especially in terms of thermomechanical stability, addition of different fillers (nanoparticles) in PLA was explored. The principal drawbacks of such a biodegradable polymer in terms of industrials application like food packaging are its poor thermal resistance, low mechanical and limited gas barrier properties. These drawbacks could be overcome by improving the thermomechanical properties through copolymerization, blending, and filling techniques. However, the use of fillers appears to be the most attractive approach because of lower cost. There are different approaches for the preparation of PLA nanocomposites: in-situ polymerization, solution intercalation, and melt intercalation. Since melt intercalation provides more advantages as compared to others, this technique has been used as a standard method to develop polymer-layer silicate nanocomposites. Use of wollastonite in high fraction will reduce the cost of composite and improve tensile strength, impact properties and dimensional stability and yield. High aspect ratio resulting on these wollastonite composites to resist machining and thus has greater surface area, better stress propagation. Reinforcement with wollastonite increases the starting crystallisation temperature and induces a shorter processing time in injection moulding and thus the effect of crystallinity of the composite for this reason the reinforcement of rotational moulded articles with wollastonite is of interest for research. These materials exhibits increase in flexural modulus, HDT, superior dimensional stability, reduced cost and ease processability. The aim of this work is to improve melt viscosity, thermo-mechanical and gas barrier properties of PLA by mixing it with aerosol/ wollastonite, so that properties able to enlarge the PLA application fields. In the present research we zero in on the PLA acid was dispersed with aerosol and wollastonite separately with different weight ratios on characterization.

II. METHODS AND MATERIAL

Poly (lactic acid) was supplied by Nature Works 4060D, and was used as a matrix. SLS and chloroform (Merck, Germany) were used as a surfactant and solvent, respectively. The grade of wollastonite (W) used for preparing different compositions was Fillex-11AB3 (surface treated), supplied by Wolkem India Limited. Aerosil (grade RY50, BET Surface area (m2/g): 30 + 15, PH: 4.5-7.5, Carbon content [wt. %]: 3.0-4.0, Chemical composition: Sio2) filler. Tensile strength, three point bending tests were carried out at par with ASTM D 53455. Tensile and flexural tests were performed on Instron universal testing machine (3369).

Synthesis of PLA composite material

Two different systems were prepared such as aerosil dispersed into PLA matrix as a first system and wollastonite particles dispersed into the same matrix for preparing the second system. PLA granules are taken in a conical beaker by 2wt.% which is mixed with 100ml of chloroform solution. This modified solution was thoroughly stirred for about 3/4hour. Once the solution is completely made then aerosil particles by 0.1wt.% dispersed into the solution. Then using spatula we stirred the complete solution for about ½ an hour. This solution was poured on to the glass mould which is prepared by ASTM standards. Make sure these mould were coated with polyvinylalcohol solution as it is facilitate easy removal. Then the specimen is allowed to cure for 24 hours and post cured composites were cut in to dumbbell shapes for testing. After doing this specimen the above procedure is repeated for getting remaining specimens by taking other aerosil weight ratios such as 0.1, 0.2, 0.25, 0.3 . Neat PLA composites were also prepared along with aerosil filled composites. In order to prepared second system wollastonite was used as a filler to disperse into the same matrix as mentioned above. ASTMD543-87 was used to prepare the specimens for conducting chemical resistance. Three acids, three alkalis and four solvents were used for this purpose.

Acetic acid, nitric acid, hydrochloric acid, ammonium hydroxide, aqueous sodium carbonate, aqueous sodium hydroxide, carbon tetrachloride, benzene, toluene, and distilled water were used after purification. In each case, the samples (5mm x 5mm x 3mm) were pre-weighed in a precision electrical balance and dipped in the respective chemical reagents for 24 hrs.

III. RESULTS AND DISCUSSIONS

Table 1: Experimental results of chemical resistance test for system-

Filler loading(wt.%)	**0.0**	**0.1**	**0.2**	**0.25**	**0.3**
Hydrochloric acid, *HCl(10%)*	1.202	1.304	1.018	0.985	0.856
Acetic acid , (CH_3COOH)(5%)	1.023	0.365	0.142	0.523	0.632
Nitric acid ,(HNO_3) (40%)	2.014	1.542	1.325	1.856	2.745
Sodium hydroxide , (NaOH) (10%)	3.421	1.892	1.754	1.458	2.563
Sodium Carbonate - ,(Na_2CO_3) (20%)	-0.235	-0.364	-0.963	-0.478	-0.421
Ammonium Hydroxide ,(NH_4OH)(10%)	0.230	0.125	0.352	0.412	0.478
Benzene	4.456	5.563	6.589	8.754	10.235
Toluene	2.365	3.012	1.025	4.023	1.002
Carbon tetrachloride, CCl_4	0.123	0.365	0.441	0.336	0.222
Distilled water,H_2O	1.023	1.089	1.005	1.456	1.025

They were then removed and immediately washed in distilled water and dried by pressing them on both sides with a filter paper at room temperature as described elsewhere. These samples were then re-weighed and the percentage loss/ gain were determined using the equation:

$$\% \, Weight\ loss\ of\ gain\ of\ the\ sample = \frac{Original\ weight - Final\ weight}{Original\ weight}\ x100$$

Table 2 : Experimental results of chemical resistance test for system-2.

Filler loading(wt.%)	**0.0**	**0.1**	**0.2**	**0.25**	**0.3**
Hydrochloric acid, *HCl(10%)*	2.102	3.402	4.014	0.865	0.582
Acetic acid ,(CH_3COOH)(5%)	1.243	0.235	0.245	0.532	0.458
Nitric acid ,(HNO_3) (40%)	2.452	1.752	1.005	1.744	2.745
Sodium hydroxide , (NaOH) (10%)	4.253	3.256	2.025	1.489	3.477
Sodium Carbonate - ,(Na_2CO_3) (20%)	0.245	0.356	0.588	0.856	0.563
Ammonium hydroxide ,(NH_4OH)(10%)	0.563	0.255	0.374	0.466	0.856
Benzene	4.475	5.744	6.889	8.452	10.253
Toluene	-2.253	-3.035	-1.025	-4.004	-1.042
Carbon tetrachloride, CCl_4	0.124	1.023	2.036	0.356	0.455
Distilled water,H_2O	1.145	1.745	1.454	1.889	1.470

Tensile strength and modulus

Table 3: Experimental results of tensile strength and modulus of system-1 and system-2.

Filler Fractions	Tensile strength (MPa)		Tensile Modulus (MPa)	
(wt.%)	System-1	System-2	System-1	System-2
0.0	5.20	5.20	1523.96	1523.96
0.10	6.06	7.23	1598.44	1835.02
0.20	6.77	8.88	1635.75	1956.33
0.25	7.11	9.56	1705.48	1985.04
0.30	6.85	8.52	1809.25	2304.63

From the above **Table 3:** Shows the experimental results of the mechanical properties such as tensile strength and modulus of system-1(PLA + aerosil) and system-2 (PLA + Wollastonite). In this study we have considered five different variations (0.0, 0.1, 0.2, 0.25, and 0.3) of filler fractions have been considered for this study. Aerosil and wollastonite are two fillers have considered for this research work. Tensile strength for system-1 and system-2 graphs were shown in the **Figure 1**. It was noticed that tensile strength was gradually increases from sample 0.0 variations to sample 0.25 variations. The tensile strength was optimized at the 0.25 variation of aerosil filler and at this variation strength was measured as 7.11 MPa, on other hand strength was decreased for the sample 0.3 variation. The reason might be due non-uniform distribution of particle or increased viscosity of the modified solution might be the reason. Even for the system-2 also similar results were noticed line strength was gradually increases from the sample 0.0 variation to till 0.25 sample variation and at the 0.25 variation performance was 9.56MPa. System-2 sample 0.25 variation has got 34.45% of increase in tensile strength was observed when compared with the system-2. Tensile modulus graphs for system-1 and system-2 were shown in the **Figure 2**. It was noticed that tensile modulus was gradually increases from sample 0.0 variation to sample 0.3 variations. The tensile modulus was optimized at the 0.3 variation of aerosil filler and at this variation strength was measured as 1809.25 MPa. When the particles are consolidated in the matrix stiffening effect will be gradually increases some extent, this might be the reason increased modulus. Even for the system-2 also similar results were noticed line strength was gradually increases from the sample 0.0 variation to till 0.3 sample variation and at the 0.3 variation performance was noticed as 2304.63 MPa is the maximum performance. In fact in system-2 sample 0.3 variation has got 27.38% of increase in tensile modulus was observed when compared with the system-2.

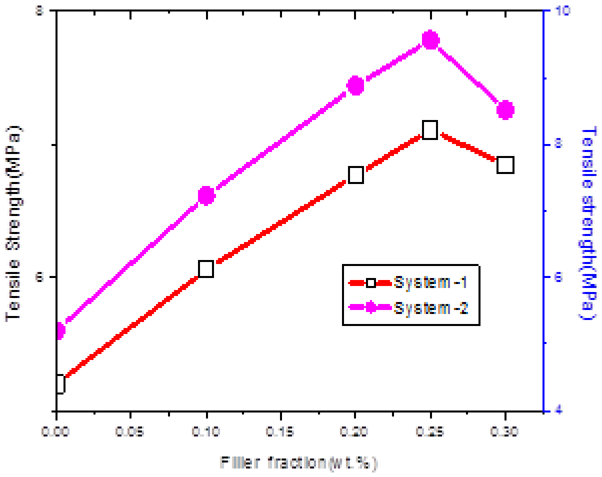

Figure 1: Variation of tensile strength performance as a function of filler fractions of two systems

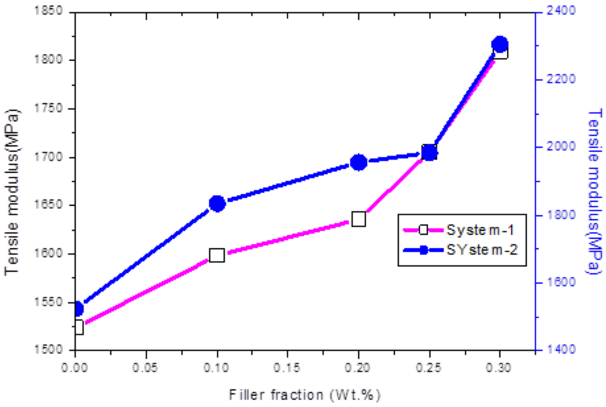

Figure 2: Variation of tensile modulus performance as a function of filler fractions of system-1 and system-2

Table 1 shows the tabulated results of the chemical resistance test for the system-1. It was observed from the results that except sodium carbonate lost weight whereas remaining all samples got some weight. Among the samples that have got weight, benzene got high weight gain than other. Loss of weight for sodium carbonate is due to the chemical reaction of the carbonated molecules with the aerosil or lactic acids. Ashok kumar et al., were observed by the similar results in their research. Moving on to **Table 2** depicts the chemical resistance results of the system-2. It was observed from the results that except toluene lost weight whereas remaining all samples gained some weight. Among the samples that have got weight, benzene got high weight gain than other. Loss of weight for toluene is due to the chemical reaction of the toluene molecules with the wollastonite particles.

IV. CONCLUSION

Aerosil/wollastonite filled PLA composites prepared with different weight ratios such as 0.1,0.2, 0.25 and 0.3 wt.%. System -1(i.e. PLA filled with aerosil filler) tensile strength was poor when compared with system-2 (PLA filled with wollastonite filler). Reasons attributed that due to high specific volume for aerosil was made the non-homogeneous mixing of the filler throughout the matrix consequently filler concentrated on the top surface identified for poor performance. On other hand, for system-2 due to low specific volume particle of these spreads all over the matrix which made uniformly distribution of the filler. For system-1 tensile strength was optimized at 0.2wt.% whereas for system-2 tensile strength was optimised at 0.25wt.%. Tensile modulus for the system-1 was increased up to 0.25wt.% on other hand tensile modulus was increased for system-2 up to 0.3wt.%. It was also noticed that chemical resistance was also found good for all the chemicals except sodium hydroxide and toluene for system-1, whereas for system-2 is also good chemical resistance except toluene.

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