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Paper

Development and Characterization of Oyster Shell Powder Filled Polypropylene Composite

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ABSTRACT: Utilizing waste materials in making useful products is a globally increasing trend. This can reduce the cost and environmental issues. In this study, oyster shell powder (OSP) is reinforced as a filler in polypropylene (PP) matrix to make a new composite. The purpose is to improve fire retardant properties of PP as a result of OSP reinforcement. Mechanical, fire retardant and water absorption properties of the new composite have been studied in this research. Concentration of OSP reinforcement in PP has been varied by wt% and its effect on the above mentioned properties has been observed. SEM (scanning electron microscopy) images of tensile and bending fractured surfaces have been taken to observe the failure mechanism during mechanical tests. An increase in the fire retardancy has been observed as a result of the OSP reinforcement while tensile strength decreased. Stiffness also increased with the addition of OSP in PP.

Key Words: Oyster shell, Fire retardant, Polypropylene, Biodegradable, Composites

1. INTRODUCTION

Some sea animals have hard protective outer shells around them. These shells are composed of excessive amount of calcium carbonate crystals together with layers of viscoelastic proteins, these dense structures exhibit excellent mechanical properties [1]. The Pacific oyster, is an oyster native to the Pacific coast of Asia, oyster can easily be found as waste material in Korea. This waste material with good mechanical properties can be utilized as bio filler in making composites. Being a biodegradable agent it can be a good option to be combined with light weight polymer matrices. Currently one of the key research area in the field of composite materials is to increase the fire resistance of these materials. Oyster shell contains 96% of calcium carbonate ($CaCO_3$) [2], which makes it a strong agent against fire. Calcium carbonate is known as a non-combustible chemical [3], which can induce fire retardancy in the polymer based composites. Since major component of the oyster shell is calcium carbonate, which gives carbon dioxide (CO_2) on decomposing after heating/burning. Carbon dioxide does not support combustion. More addition of oyster shell powder produces more CO_2 because of higher concentration of calcium carbonate. Hence there will be a low tendency of composite to burn as CO_2 is a good fire extinguisher.

Polypropylene (PP) is a light weight polymer matrix and the fillers may increase its stiffness which has an advantage in the high temperature applications [4]. Major advantages of PP based composites include low price, light weight and versatility. PP is weak against fire and thermal loads. In applications where there is a risk of fire or heat, PP needs to be reinforced with such fillers which can improve its fire retardancy and stiffness.

Research studies on some sea shells powders have already been published in literature [1,5,6]. The researchers have studied the effect of the concentration and particle size of the filler material on the strength and fire retardant properties of the final composite. However oyster shell filled polypropylene composite has not been reported in literature yet. The purpose of this research is to develop a fire retardant waste material composite for various applications like automobiles, electronic

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insulations and packaging, etc. In such applications, fire retardancy is a primary concern. A study on strength, fire retardancy and water absorption properties of the oyster shell powder reinforced PP has been carried out by performing different types of mechanical, flame and water absorption tests. It is observed that oyster shell powder has improved the burning properties of the composite on the cost of a little decrease in mechanical strength. The optimized concentration of oyster shell powder has been suggested after the study however it depends upon the type of application. Slight decrease in the mechanical properties is common in particulate filled composites [7] but the main benefit of the oyster shell powder over other fillers is its easy availability, biodegradability and low cost which is almost free because it's a waste material.

2. MATERIALS AND METHODS

2.1 Composite Preparation

The composite specimens have been manufactured using injection molding technique. Amount of the oyster shell powder varies as 0~50 wt% of the weight of PP, particle size of the powder was 3 μm. Maleic anhydride polypropylene (MAPP) was added as coupling agent between the fillers and PP. Manufacturing process of the composite was composed of four main steps. Initially both the constituents were mixed together and shaken, then extruded to rod shapes at a temperature between 210~220 [8] with the help of a twin screw extruder. The extruded rods were cut into small pallets using a cutter. The pallets were then put into the injection molding machine and specimens of the desired shape were obtained.

2.2 Flame Test

Horizontal flame test was carried out according to ASTM D635 as shown in Fig. 1. Burning time and burning rate of each specimen have been calculated.

2.3 Tensile Test

Tensile test of the composite specimens has been performed using MTS 97 kN load cell (as shown in Fig. 1) with a head speed of 3 mm/min. Specimens geometry and experiment were setup according to ASTM standard D638 for tensile tests.

2.4 Bending Test

Three points bending test has been conducted according to the specifications recommended in ASTM standard D790 using a load cell of 5 tons manufactured by R & B Unitech M, Korea (as shown in Fig. 1) with a head speed of 3 mm/min.

2.5 Impact Test

Izod impact test was performed according to ASTM D256 using an impact hammer of 22 J. Cometech QC-639F impact testing machine was used to conduct this test.

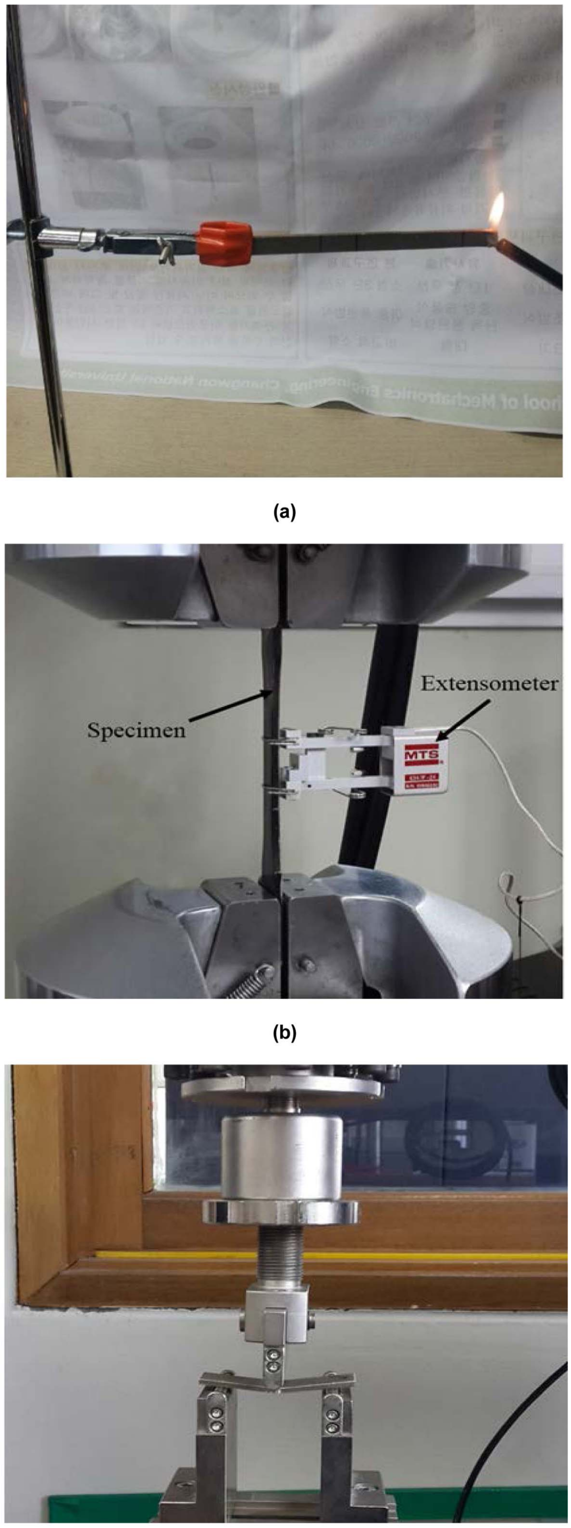

Fig. 1. Test setup for (a) flame test (b) tensile test (c) bending test

2.6 Water Absorption Test

Water absorption test was performed according to ASTM

D570. Conditioning and reconditioning were carried out at 55°C for 24 hours each. Specimens were immersed in distilled water for 24 hours.

3. RESULTS AND DISCUSSION

3.1 Burning Time and Burning Rate

The burning time and burning rate as shown in Fig. 2 have been calculated using horizontal flame test as described above. It is observed that burning time of the composite is being prolonged with the addition of oyster shell powder and ultimately burning rate is decreased. The burning time is protracted about 32% of the pure PP with the addition of 50 wt% of oyster shell, similarly burn propagation rate has been decreased by 24% of the base material PP after the addition of 50% wt of oyster shell powder.

This occurrence of decreasing flame propagation rate practically conforms that the flame retardant property of polypropylene is significantly improved. As mentioned earlier in the introduction section this reduction in the flame propagation is because of the carbon dioxide produced during the burning process of calcium carbonate.

3.2 Tensile Strength and Modulus

The effect of oyster shell powder concentration on tensile properties of the composite has been shown in Fig. 3. On increasing the fire retardant properties of the composite there is a little sacrifice of tensile strength. It's is observed that tensile strength of the composite is reduced slightly with the addition of oyster shell powder as filler. This phenomenon is common with particulate fillers in literature. At the same time tensile modulus is witnessed to be improved as a result of the addition of oyster shell content. This increase in modulus is an evidence of stiffness being induced in the material with the addition of filler. Decrease in the tensile strength is undesirable but it is not decreasing consistently rather the value of tensile strength

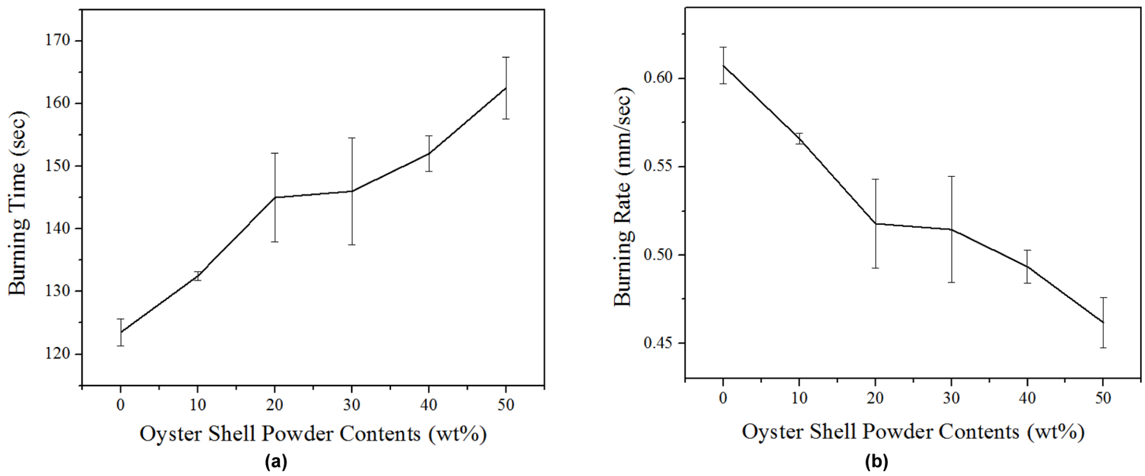

Fig. 2. Effect of oyster shell powder on (a) burning time (b) burning rate

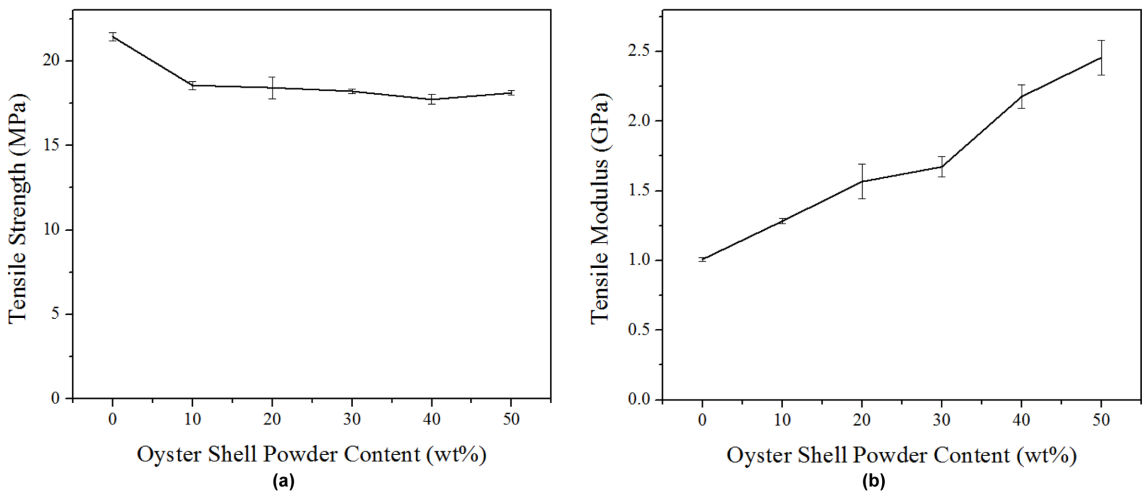

Fig. 3. Effect of oyster shell powder on (a) tensile strength (b) tensile modulus

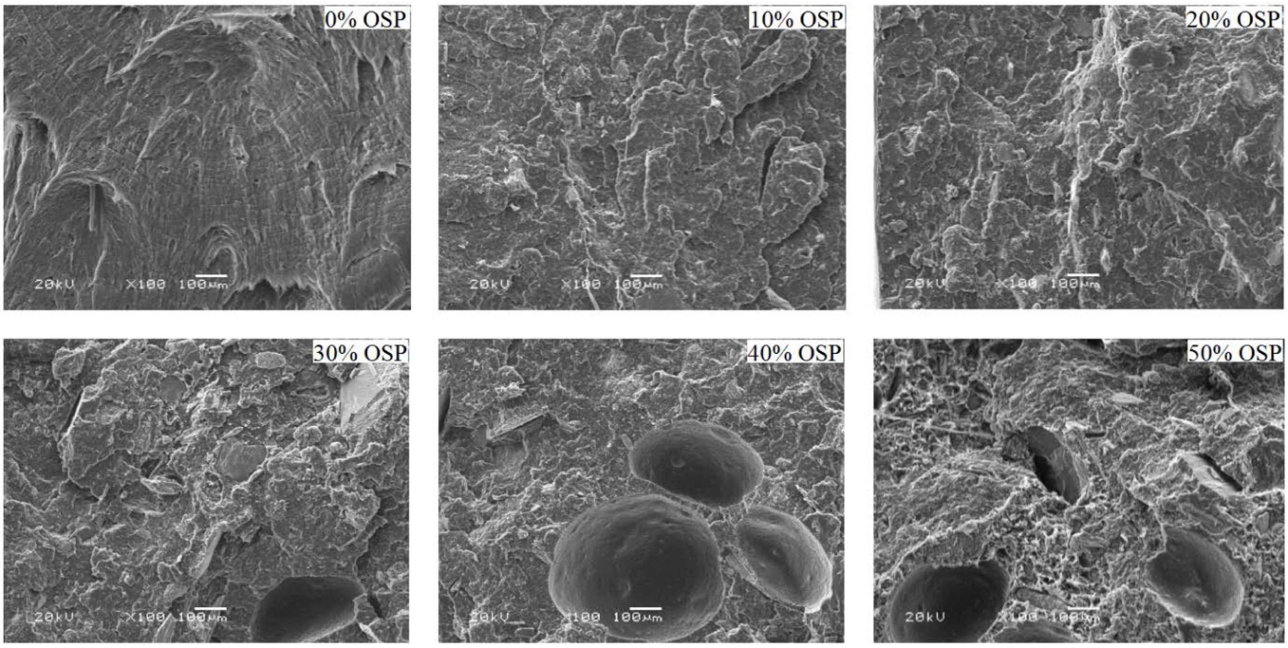

Fig. 4. SEM images of the OSP/PP specimens after tensile fracture

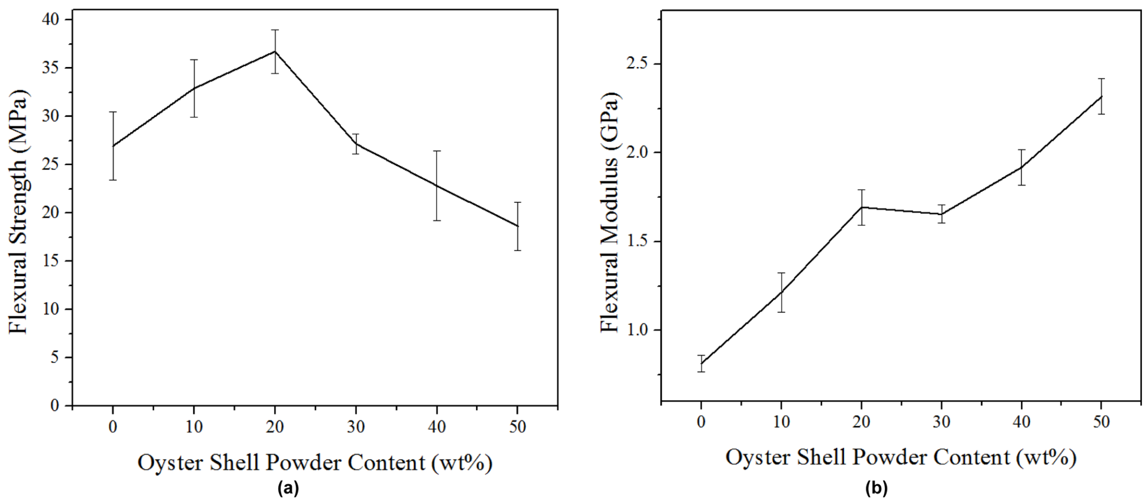

Fig. 5. Effect of oyster shell powder on (a) flexural strength (b) flexural modulus

is observed to be near stable after the first fall as shown in Fig. 3(a). Decrease in the tensile strength is about 15% and increase in the modulus is significantly high, which is about 143% as a result of the addition of 50 wt% oyster shell powder. This decrease in tensile strength is a fact as powder fillers are week against tension because of the poor interfacial bonding between the filler and matrix particles.

Fig. 4 shows SEM images of the tensile fractured surfaces of the composites. SEM images revealed the failure mechanism of OSP/PP composite. It can be observed that the failure of pure PP specimen is a ductile fracture while OSP filled PP specimens failed in a brittle way. This brittleness increases with the increasing concentration of OSP in the specimens. Moreover poor interfacial bonding between OSP particles and matrix is also a reason of fall in the mechanical properties.

3.3 Flexural Strength and Flexural Modulus

Flexural strength and modulus have been calculated through the three points bending test as described earlier, the results are shown in Fig. 5. A very interesting behavior has been noticed as a result of the flexural test on the basis of which it is easy for us to choose the optimum concentration of the oyster shell powder as filler in PP. It is witnessed that flexural strength of the composite is increasing initially with the addition of oyster shell powder content, this occurs till 20 wt% concentration of the filler but then starts falling again when

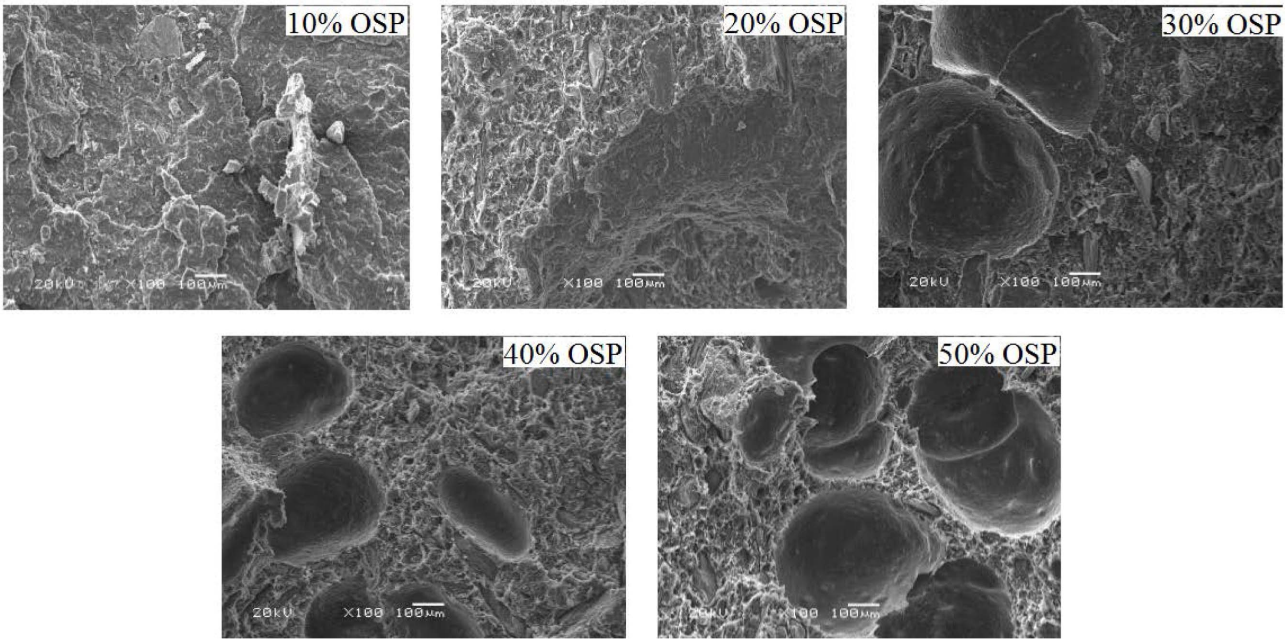

Fig. 6. SEM images of the specimens after bending fracture

the filler powder is continuously added beyond 20 wt%. On the other hand flexural modulus is also increasing as a result of oyster shell powder addition, which is again a sign of stiffness. Maximum increase noticed in the flexural strength is 36% of the pure PP value.

Fig. 6 shows SEM images of the flexural fractured surfaces of the composites. SEM images show that specimens with OSP concentration more than 20% have poor interfacial bonding between matrix and powder particles. This is the reason why these specimens have low flexural strength. Particulate filled materials usually show better performance under compression as compared to tensile loads, therefore initially specimens under bending loads showed good strength till 20% OSP reinforcement. The strength started decreasing again beyond 20% OSP filled specimens, which shows that 20% OSP reinforcement is optimal in case of bending loads.

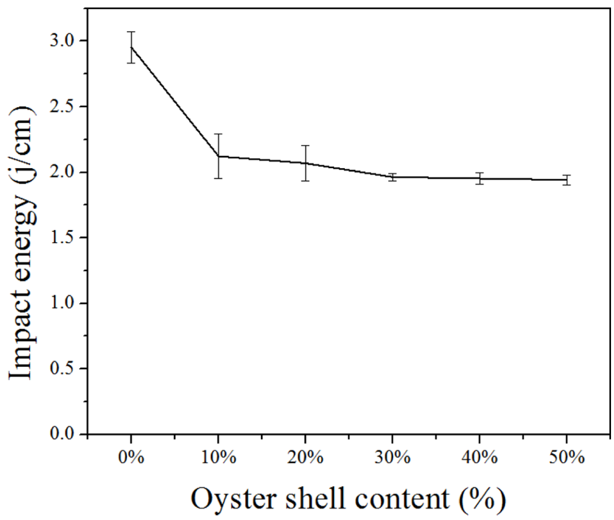

Fig. 7. Impact energy variation as a result of OSP concentration

3.4 Impact Strength

Izod impact test of the notched specimens was conducted according to the specifications mentioned in section 2.5. Results of the impact test are shown in the Fig. 7. The results suggest that the impact strength of composite specimens is decreasing as a function of the concentration of OSP. It's obvious and is correlated with the tensile test results. In the tensile test results above, we learned that stiffness of the material is increased at high concentration of OSP in PP. In this way material is becoming more brittle and hence impact strength is decreasing. Similar to the tensile test results, a prominent initial fall in the impact strength values is observed when 10% OSP was added to PP. With more addition of OSP beyond 10%, the fall in the values of impact strength is little. Poor interfacial bonding and presence of pores is also a reason of decrease in the impact strength values.

3.5 Water Absorption and Soluble Matter Loss

Water absorption has been estimated from the increase in weight of the composite after 24 hours of immersion in distilled water and soluble matter lost has been calculated after reconditioning of the specimens in oven. The results are shown in Fig. 8. As obvious the weight of the composite is

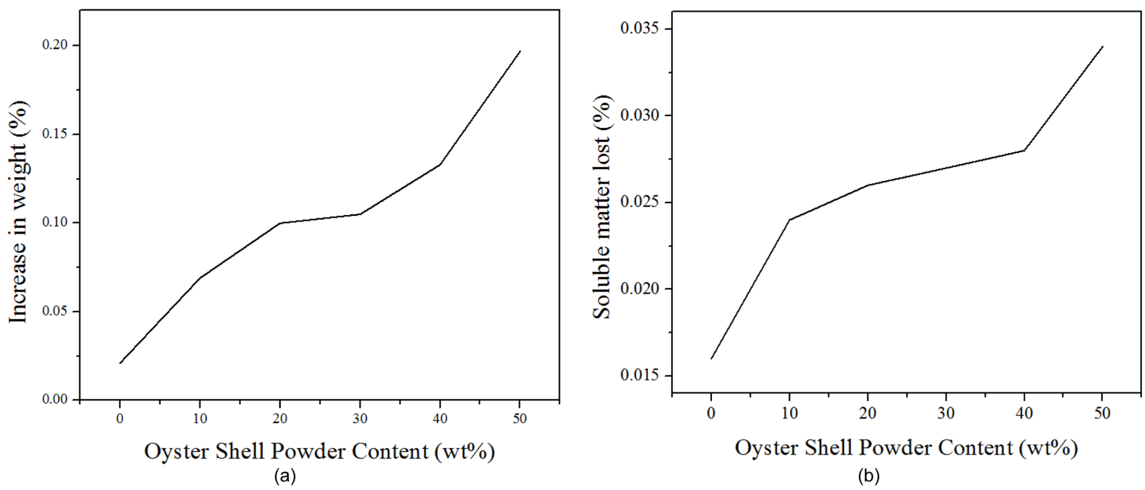

Fig. 8. Effect of oyster shell powder on (a) water absorption (b) soluble matter lost

increased after water absorption but the increase is very small which 0.2% for the 50 wt% oyster shell powder reinforced PP specimens. Similarly the soluble matter lost is also negligibly small, which is 0.034% in case of the 50 wt% oyster shell powder filled PP specimens. These small values indicate that the composite is good against water.

4. CONCLUSION

The aim of the study was to induce fire retardancy in the composite which is successfully achieved. It is observed that burning time of the composite is prolonged with the addition of oyster shell powder to the PP resin. This increase in burning time and decrease in the burning rate practically show an improvement in fire retardant property of the composite. At the cost of the improvement in flame retardancy, there occurred a slight decrease in tensile strength. The decrease in the yield strength is not consistent with the increasing concentration of oyster shell powder which is a good sign. Flexural strength is observed to increase till 20 wt% concentration of oysters shell powder, which can be taken as optimum amount of the filler in applications where bending and fire retardancy are important at the same time. Modulus is observed to increase with the increase in the oyster shell powder concentration, which means the composite is becoming stiffer with the addition of the powder. Water absorption capability of the composite is also very low, also the solubility of particles in water is very small, it shows the material is strong against water.

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