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Effect of particle size on the properties of Polyester/Palm Kernel Shell (PKS) Particulate Composites

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Abstract
The aim of the present study was to develop polyester/ palm kernel shell (PKS) particulate composites and to investigate the effect of particle size on the properties of the composites. The palm kernel shell particles were varied thus; 0, 10, 20, 30 and 40wt% at three different particle sizes; 75µm, 150µm and 300µm. Cobalt accelerator and Methyl-ethyl Ketone catalyst were utilized to initiate polymerization reaction and thus speed up the reaction. The effect of palm kernel shell particles and particle size on mechanical and physical properties of polyester was studied. The results showed a better interaction of polyester and palm kernel shell particles at 300µm sieve size with density, water absorption, ultimate tensile strength and impact energy increasing upon increase in percent palm kernel shell particles with only hardness decreasing upon increase in percent palm kernel shell.

Keywords: Palm Kernel Shell, Polyester, Particle size, Composites, Water absorption.

1. Introduction
The development in science and technology required a variety of polymer with good properties and low cost. Therefore, polymer composites were considered to be among the more promising approaches to yield new materials and have been investigated extensively. In recent years, many studies have been dedicated to utilize lignocellulosic fillers such as coconut shell, wood, pineapple leaf, palm kernel shell, etc. as fillers in order to replace synthetic fillers through utilization of natural fillers or reinforcement in thermoplastic and thermoset polymer composites in an attempt to minimize the cost, increase productivity and enhance mechanical properties of product [1]. Lignocellulosic materials as reinforcing fillers in plastics, in place of the previously used inorganic substances and synthetic fibers, offer a major benefit in terms of environmental protection [2]. The benefits offered by lignocellulosic materials over synthetic fibers like aramid, carbon or glass fiber are low densities, non abrasive, non-toxic, high filling levels possible resulting in high stiffness and specific properties, biodegradable, low cost, good thermal and acoustic properties, good calorific value and enhanced energy recovery [3]. More importantly, lignocellulosic-based fillers are derived from renewable resources [1].
Extensive studies on the preparation and properties of thermosetting and thermoplastic composites filled with jute [4], sisal [5-6], coconut shell [7-8], coir [9], bagasse [10], Rice-husk [2] etc.have beeninvestigated.
The use of biomaterials in general and agro-waste in particular is a subject of great interest nowadays not only from the technological and scientific points of view but also socially and economically in terms of employment, cost and environmental issues.
Nigeria is endowed with a lot of mineral and agro-based resources including Palm oil from Palm Kernels that could be used in the development of environmental- friendly composite materials such as Eco-pad used in modern vehicle braking systems. Food Agriculture Organisation (FAO) data showed production increased by over 400% between 1994 and 2004, to over 8.66 million metric tones. In 2008, Malaysia produced 17.7 million tonnes of palm oil on 4,500,000 hectares of land and was the second largest producer of palm oil, employing more than 570,000 people. Malaysia is the world's second largest exporter of palm oil after Indonesia. As at 2011,

Nigeria was the third-largest producer with more than 2.5 million hectares (6.2×106 acres) under cultivation. Until 1934, Nigeria had been the world's largest producer [11].
Palm kernel shell (PKS) is the hard endocarp of palm kernel fruit that surrounds the palm seed. It is obtained as crushed pieces after threshing or crushing to remove the seed which is used in the production of palm kernel oil [12]. Furthermore, they are waste materials that are normally stockpiled in open fields thereby subjecting them to varying climatic conditions [13]. The shells are flaky and of irregular shape that depend on the breaking pattern of the nut. The shell is made up of 33% charcoal, 45% pyroligneous liquor and 21% combustible gas [11]. Oil palm fibers have been extensively studied for the production of various composites, such as thermoplastic composites, particleboard, medium density fireboard polymer impregnated oil palm trunk and other thermoset composites [14].
Some of the areas where palm kernel shell are used or are being considered for use include: automobile disk brake pad, carbon activation for water purification, concrete ingredient in building industry, fuel for heat generation, thermal insulator etc. [11]
As society begins to recognize the importance of utilizing renewable bioproducts that are beneficial to the environment, focus is beginning to return to agricultural materials [15]. Agricultural wastes are recently researched into in great extent to: reduce the problem of waste disposal, produce much cheaper materials, increase the use of waste materials and make use of a more available material with little or no processing and low production cost.
In the light of the above, the present study is channeled towards producing a composite material using a thermoset polymer (Unsaturated polyester) as the matrix and a lignocellulosic material (Palm Kernel Shell (PKS) particulates) as the reinforcing filler so as to furtherestablish the use of agricultural wastes as reinforcements in polymer matrices. Also the study tends to use mechanical and physical properties as criteria in establishing the possibility of using lignocellulosic materials as reinforcing fillers in thermoset polymers.

2. Materials and Methods

2.1. Materials

The materials used in this research were: Palm kernel shell (PKS) obtained from Old Kulo, Kuje area council, Abuja, Nigeria, liquid Polyester resin, Cobalt accelerator (purple colour) and Methyl-ethyl Ketone catalyst (colourless) which were obtained from central market, Kano road, Kaduna State, Nigeria.

2.2. Sample Preparation

The PKS was washed thoroughly with water and detergent to remove dirts, dried in the sun for 2 days after which it was ground in a hammer mill to particles. The particulate was then sieved into different sieve sizes of 75µm, 150µm and 300µm. For each sieve size of PKS, the weight percent of 10, 20, 30 and 40 were used for the production of the composites with Polyester as the matrix. The accelerator and the catalyst were added in 10% by volume of the overall volume of Polyester and PKS. The composites were produced by mixing the polyester with the accelerator followed by the catalyst before the addition of the PKS. The mix was then poured into a mould and allowed to set. The composites were further cured by heating in an oven at 80^{o}C for 3hours. The composites were cut into the required test sizes of Tensile, Impact, Hardness and water absorption.

2.3. Density Determination

The densities of PKS, polyester and the composites were determined by obtaining their weights and volumes and using equation 1 for computation.

$$Density = \frac{Mass}{Volume} (g/cm^3) \quad (1)$$

2.4. Water Absorption Test

Certain weights of the composites were cut off and weighed (W_1) before immersing in water for 2 days. The samples were then removed from the water, cleaned and dried with blotting paper and weighed again (W_2). The weights of moisture absorbed by the composites were calculated by the formula:

$$\% \text{ Moisture Absorbed} = \frac{W2-W1}{W1} \times 100 \quad (2)$$

2.5. Chemical Composition of Palm Kernel Shell

The chemical composition of the PKS was determined using the fibertech compartment at central laboratory services unit in National Animal Production Research Institute (NAPRI), Shika, Nigeria.

2.6. Determination of Tensile Properties

Tensile tests were carried out using the Hounsfield tensometer with serial No.W3179 by placing the edges of the samples in between the grips of the machine before applying the load increasingly until the sample fractures. The pricker was used to obtain the readings of the load and extension on a graph sheet wrapped round an autographic recording drum which rotates as the load is applied on the sample. The ultimate tensile strength (UTS) and percentage elongation were obtained from the load/extension curve for each sample.

2.7. Hardness Values Determination

The hardness values of the samples were obtained using the Indentec universal hardness testing machine, 8187.5 LKV model B. The test type was Rockwell hardness with scale F (HRF), the indenter was a 1/16 inch steel ball, the minor and major loads were 10 and 60 Kgf respectively. The samples were placed on the anvils and the minor load was applied to the samples and then zero datum position was established and then the major load was applied. This loading process was repeated on three different positions on each sample and the average value was obtained from the digital display on the machine as the hardness value of each sample.

2.8. Impact Energy Determination

Charpy impact testing machine (capacity: 15 and 25 Joules) was used to obtain the impact energy values of the composites. The pendulum was raised to the test height and held there. The sample was mounted in the machine and the door of the machine was closed. The pointer for reading the impact energy value on the calibrated scale was adjusted to zero before the pendulum was released by means of a handle on the door of the machine. The pendulum falls from the height, breaking the sample and hitting the pointer to the test energy value.

3. Results and Discussion

3.1. Density

The results for the density determination are shown in Table1 and Figure 1 for the various sieve sizes and compositions of the composites produced.

Table 1: Density values of the composites produced (density of PKS is 2.50 g/cm³).

% Composition of PKS	Density (g/cm³)		
	75 µm PKS	150 µm PKS	300 µm PKS
0	1.104	1.104	1.104
10	1.158	1.205	1.200
20	1.161	1.236	1.211
30	1.167	1.341	1.405
40	1.408	1.338	1.395

The density values as shown in Figure 1 generally increase as the percentage composition of PKS increase. This could be expected as the density of PKS obtained (2.50g/m^3) is greater than that of Polyester (1.014g/m3). Furthermore, it was observed that as the particle size of PKS increases, the density values also increased. Similar observation was reported by Dagwa et al. [11] who observed that density values increase with increase in particle size of PKS and compares favourable with the densities of sawdust particle sizes. They attributed such behavior to the fact that for smaller particle sizes, their compressibilities were higher because they had more porosity. Also, Husseinyah et al. [16]observed that the density of coconut shell filled polyester composites increased with increase in filler content.

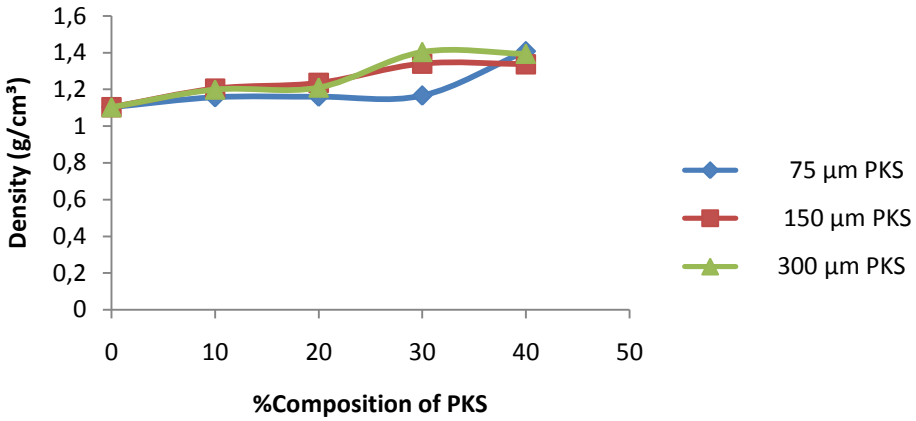

Figure 1: Variationof density with percentage composition of PKS

3.2. Water Absorption

The results obtained for the water absorption test of the composites after immersion for two days are shown in Table 2and Figure2.

Table 2: Water absorption values of the various composites produced.

% Composition of PKS	%Water Absorption		
	75 µm PKS	150 µm PKS	300 µm PKS
0	0.092	0.092	0.092
10	1.76	5.04	1.57
20	7.83	1.56	1.95
30	1.53	2.06	1.80
40	2.13	1.06	2.46

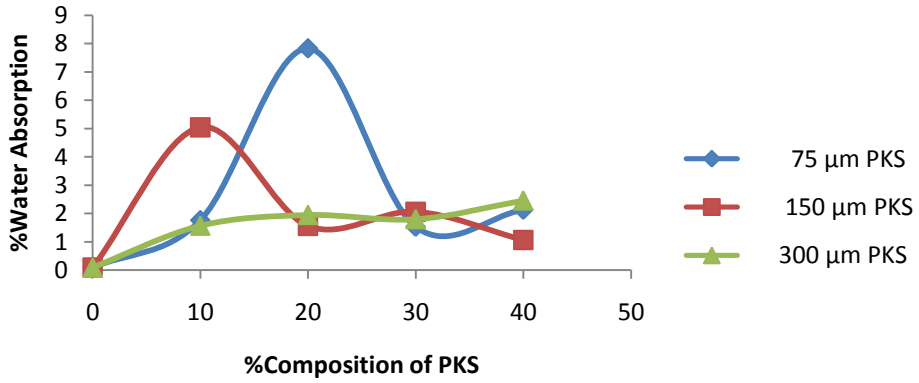

Figure 2: Plot of water absorption against %composition of PKS

For each particle size, there were variations in water absorption behavior as composition of PKS increases. For the 75µm sieve size, the maximum value was obtained at 20wt% PKS; that of 150µm sieve size was obtained at10% PKS with a value of 5.04% while the maximum for the 300µm sieve size was 2.46% at 40% PKS.It is an established fact lignocellulosic materials are hydrophilic in nature since their main constituents are cellulose, hemicellulose, lignin and others which is a factor that contributes to their absorption of moisture from the atmosphere [17]. From Figure 2, it was observed that the 300µm sieve size of PKS had the least amount of water absorbed after two days of immersion. This agrees with the observations of Dagwa et al [11] who reported that

the smaller the particle size, the more the moisture sorption and they concluded that the behavior could be attributed to the increase in surface area and inter–particle friction leading to smaller flow rates of the particles.

3.3. Chemical Composition of PKS

The chemical composition of PKS as determined from the fibertech compartment at the National Animal Production Research Institute(NAPRI) Shika, Nigeriais given in Table 3below:

Table 3: Chemical composition of PKS (result obtained from NAPRI).

Property	Moisture content	Ashcontent	Lignin	Cellulose
% composition	5.55	2.35	44.47	26.65

Figure 3: Fibertech Compartment, NAPRI used for the determination of the PKS chemical composition.

3.4. Tensile Properties

Tables 4 – 6 show the tensile properties of the composites and Figure 4 shows the tensile strength of the composites produced.

Table 4: Tensile properties of polyester/PKS composites with PKS of 75µm sieve size.

% Composition of PKS	Max. Load (N)	%Elongation	U. T. S. (N/mm^2)
0	1035	12	18.19
10	666.6	10.13	18.74
20	777.0	10.31	17.58
30	450.0	8.85	18.62
40	744.6	9.48	18.79

Table 5: Tensile properties of polyester/PKS composites with PKS of 150µm sieve size.

% Compositionof PKS	Max. Load (N)	%Elongation	U. T. S. (N/mm^2)
0	1035	12	18.19
10	547.6	10.63	18.20
20	644.0	13.68	18.96
30	857.4	10.20	18.64
40	831.0	7.35	17.92

Table 6: Tensile properties of polyester/PKS composites with PKS of 300µm sieve size.

% Compositionof PKS	Max. Load (N)	%Elongation	U. T. S. (N/mm^2)
0	1035	12	18.19
10	641.6	9.75	18.01
20	686.9	10.43	18.97
30	817.5	10.65	19.17
40	804.2	9.9	19.21

From the results of the tensile strength of the composites produced, it was observed that there was an initial drop of strength from 18.19N/mm^2 (0% PKS) to 18.01N/mm^2 (10% PKS)for the 300µm sieve size PKS composites then increased progressively to 19.21N/mm^2 at 40% PKS addition. The composites produced with the 150µm sieve size PKS showed an increment in tensile strength up till 20% PKS addition before dropping to 17.92N/mm^2 at 40% PKS addition this is with noting the fact the strength at 30% PKS addition (18.64N/mm^2) is higher than that of the unreinforced polyester (18.19N/mm^2). The trend was quite different with the composites produced with the 75µm sieve size PKS since it showed significant increase in strength from 18.19N/mm^2 (0% PKS) to 18.74N/mm^2 (10% PKS) before dropping at 20% PKS addition after which it increasedprogressively to 18.79N/mm^2 at 40% PKS addition. However, the drop in strength value at 20% PKS addition for the 75µm sieve size of PKS composite was expected since there was a sharp increase in water absorption (see Figure 2)atthat compostion.Generally, the results are in agreement with Husseinyah et al. [16], who observed that the tensile strength of coconut shell filled polyester composites decreased with addition of 15php filler content and then started to increase with increasing filler content and Njoku et al [18], in which it was concluded that the strength increased with increase in periwinkle shell particle weight fraction from 10 – 45wt%.

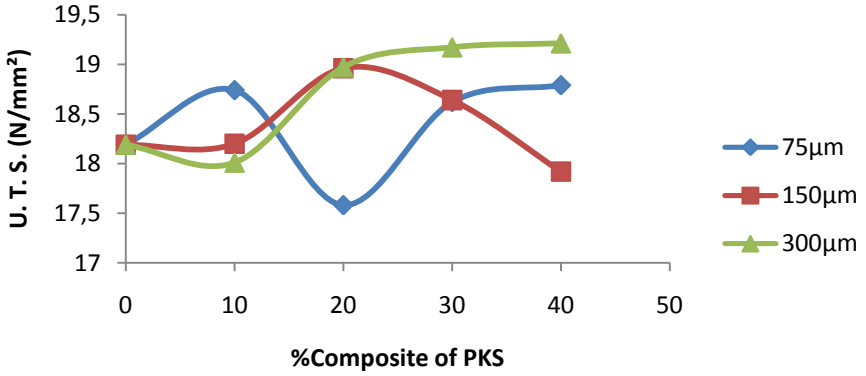

Figure 4: Plot of U. T. S. against %composition of PKS.

3.5. Hardness Values

The results for hardness values of the composites are given in Figure 5 and Table 7. In Figure 5, the general trend for our observation in hardness test was that there was decrease of hardness values for the composites produced except for some isolated cases as seen in Table 7. However, this is contrary to Ishidi et al [19]thatreinforced HDPEwith 30, 40 and 60wt% PKS and noticed that hardness values increased upon increase in PKS and then decreased at 60wt% PKS and El-Shekiel et al [20] who also reported increment in hardness values but Kumar et al [21] reported an initial increment, then decrease in hardness values of the composites they produced.

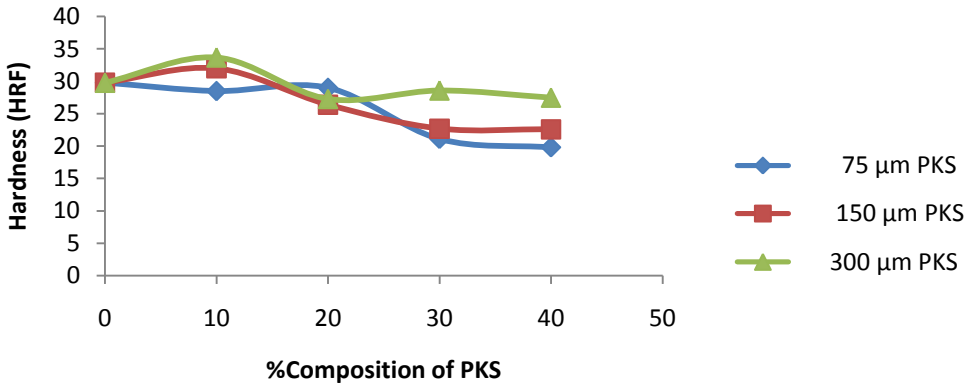

Figure 5: Plot of hardness against % composition of PKS.

Table 7: Hardness values of the composites produced.

% Composition of PKS	Hardness Values (HRF)		
	75 µm PKS	150 µm PKS	300 µm PKS
0	29.8	29.8	29.8
10	28.5	32.0	33.7
20	29.0	26.4	27.3
30	21.1	22.7	28.6
40	19.8	22.6	27.5

3.6. Impact Values

Table 8 and Figure 6 show the impact energy values of the composites produced.

Table 8: Impact energy of the composites produced.

% Composition of PKS	Impact Energy (joules)		
	75 µm PKS	150 µm PKS	300 µm PKS
0	0.200	0.200	0.200
10	0.400	0.325	0.275
20	0.275	0.275	0.325
30	0.300	0.375	0.330
40	0.375	0.325	0.345

The impact energy values increased from 0% to 10% composition for all the particle sizes. However, the increase was steady for the 300µm particle size unlike for the other particle sizes in which fluctuations were observed but noting the fact that the values were higher than that of the unreinforced polymer. The results obtained here is contrary to the work of Ishidi et al [19]; in which they discovered that the tensile-impact strength of unnotched composites decreases as filler loading increases. Kumar et al [21] also reported an increase then decrease of impact energy in their submission.

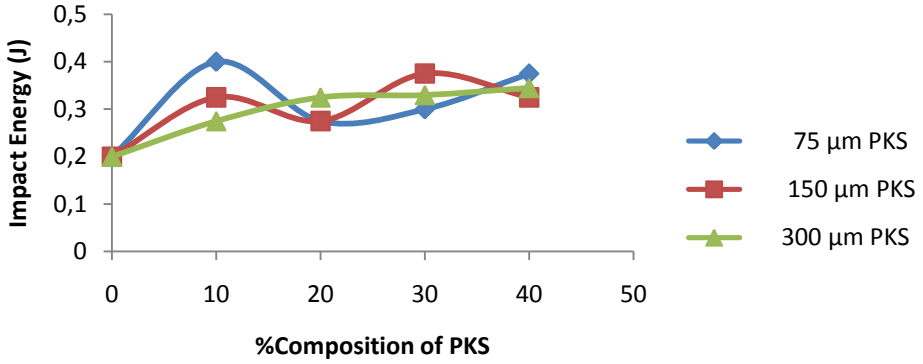

Figure6: Plot of impact energy against %composition of PKS.

Conclusion

The present study centered on the production of polyester/palm kernel shell (PKS) particulate composites and studying the effect of PKS particle size (75µm, 150µm and 300µm) on the mechanical and physical properties of the composites produced. The particulate loading was in the order of 10, 20, 30, 40wt% PKS particulates.

At the end of this study, it was established that the densities of the composites increased for all the sieve sizes under consideration with increase in weight percent of PKS particles.

Other findings at the end of the study include:

i. A general decrease in hardness values for all the sieve sizes of PKS particles with a few exceptions.
ii. The 300µm sieve size PKS/polyester composites had the most steady/definite properties investigated as compared to those of the other sieve sizes.

iii. The composites produced with the 300µm sieve size PKS gave the best tensile strength values as compared to those of other sieve sizes.
iv. The impact energy values for all the sieve sizes were greater than that of the unreinforced polymer on a general note.
v. It was observed that the 300µm sieve size PKS had better interaction with the polyester resulting in better mechanical and physical properties.

References

1. Salmah H., Marliza M., Teh P.L. s*internat.J. Engr. Technol.* 13(02) (2013)9 4.
2. Yang H-S., Kim H-J., Son J., Park H-J., Lee B-J., Hwang T-S. *comp. Struct.* 63 (2004) 305.
3. Raju G.U., Kumarappa S., Gaitonde V.N. *J. Mater. Environ. Sci.* 3(5) (2012) 907.
4. Rana A.K., Mitra, B.C., Banerjee A.N. *J. Appl. Polym. Sci.*71 (1999) 531.
5. Joseph P.V., Rabello, M.S., Mattoso, L.H.C., Joseph, K., Thomas, S. *Comp. Sci. Technol.* 62 (2002) 1357.
6. John M.J., Francis, B., Varughese, K.T., Thomas, S.*Comp. A* 39 (2008) 352.
7. Bledzki A.K., Mamun, A.A., Volk, J. *Comp. Sci. Technol.* 70 (2010) 840.
8. Bhaskar J., Singh V.K. *J. Mater. Environ. Sci.* 4(1) (2013) 113.
9. Rout J., Misra M., Tripathy S.S., Nayak, S.K., Mohanty, A.K., *Comp. Sci. Technol*. 61 (2001) 1303.
10. Saini G., Narula, A.K., Choudhary, V., Bhardwaj R.*J. Reinf. Plast. Comp.* 29 (2010) 731.
11. Dagwa I.M., Builders, P.F. and Achebo, J.*Intern. J. Mech. and Mechatr. Engr.,* 12(4) (2012) 88.
12. Olutoge F.A. *ARPN J. Engr. Appl. Sci.* 5(4) (2010) 7.
13. Alengaram U. J., Jumaat, M. Z., Mahmud, H. *J. Appl. Sci.* 8(18) (2008) 3207.
14. Olumuyiwa O.J., Isaac T.S., Adewunmi O.A., Ololade A.I. *J. Min. and MatlsCharact. Engr.* 11 (2012) 1.
15. Mohamed Nor, (2007): Development of Coconut Shell Powder Reinforced HDPE Composites, MSc., Department of Mechanical Engineering, University of Technical Malaysia, Melaka.
16. Husseinsyah S. and Mostapha, M. *Malays. Polym., J.* 6 (1) (2011) 87.
17. Mohanty A.K., Misra, M., Drzal, L.T. *Comp. Interf.,* 8(5) (2001) 313.
18. Njoku R. E., Okon, A. E. and Kpaki, T. C.*Nig. J. Technol.,* 30(2) (2011) 87.
19. Ishidi E.Y., Adamu I. K., Kolawale E. G., Sunmonu K.O., Yakubu M.K. *J. Emerg. T. in Engr. and Appl. Sci.*, 2 (2) (2011) 346.
20. El-Sheikel Y.A., Sapuan S.M., Abdan K., Zainudin E.S., *Mater. Design*, 40 (2012) 299.
21. Kumar M.A., Reddy G.R., Bharathi Y.S., Naidu S.V., Naidu N.P., *J. Comp. Mater*. 44 (2010) 3195.