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Physical and thermal characterization of natural fibre extracted from *Caryota urens* spadix fibre

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In this study, comprehensive characterization of *Caryota urens* spadix fibre has been done to examine its morphological, physical, mechanical, chemical and thermal properties. High cellulose content of the fibre (42.1 wt%) provides better tensile strength (1970-6330 MPa) and ensures better bonding with the matrix. Moreover, the low density of the fibre (0.78 $g \cdot cm^{-3}$) makes it alternative for hazardous synthetic fibres. The lower crystal size structure tends to absorb more water than the higher crystal size structure. The thermogravimetric analysis confirms its stability up to 270°C, which is higher than the polymerization temperature. The results confirm the potential of *Caryota urens* spadix fibres as reinforcement in bio-reinforced polymer composites for automotive and structural applications.

Keywords: Bio-reinforced polymer, *Caryota urens*, Natural fibre, Thermal characterization, Thermogravimetric analysis, Tensile strength

1 Introduction

New approaches for the modification of plastics properties have been explained due to growing concern about the environment and the constant search for the use of fillers in polymeric materials[1]. In recent year, the textile industry is looking for manufacturing go green products. Ultimately the *Caryota urens* spadixes are also known for obtaining ecofriendly fabrics[2]. The study of molecular structure of the lingo-cellulosic fibres enables the knowledge of their chemical structure, and therefore, justifies a better use as filler in a polymer matrix, giving rise to composites with improved properties[3]. The goal is to produce an improved and sustainable products made of fibrous material which can be used in textile industry, such as garments, upholsteries and interior decorations. Especially the plant fibre has the characteristics, such as resistance to water, thermal insulation, etc.

Thus, the presence of the new plant fibre decreases the pressure due to handful number of species available for the small scale industry[4,5]. Researchers have investigated the

physico-chemical, mechanical, and thermal properties of several new natural cellulosic fibre such as *P. juliflora* bark fibre, *Cissus quadrangularis* root fibre, *Phoenix reclinata*, snake grass fibre, *Acacia leucophloea* bark, etc. using chemical analysis, single fibre tensile testing, Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and thermo-gravimetric analysis (TGA) and SEM analysis. The recently identified *Caryota urens* Spadix fibre extracted from *Caryota urens* plant spadix has not been investigated for its physico-chemical, mechanical, and thermal properties till now. The *Caryota urens* plant belongs to the family Arecaceae, native to tropical and subtropical regions and sometimes cultivated as an ornamental. The root, bark, flowers, leaves, and seed of *Caryota urens* plant are being used for sugar preparation.

Concerning the above, this investigation deals with the extraction of *Caryota urens* fibre from the spadix of *Caryota urens* plant and analysis of the physico-chemical, mechanical, and thermal properties of this fibre using XRD method, FTIR spectroscopy, TGA, and single fibre tensile as well as its comparison with other natural fibres.

2 Materials and Methods

2.1 Materials

Caryota urens Spadix were collected from Coimbatore district, Tamil Nadu, India. Flowering begins at the top of the trunk and often continue moving downwards for several years. Individual staminate flowers remain open for 16-20 days, while a

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single inflorescence has flowers opening for about 6 weeks. This is generally found in India, Malaysia, Myanmar, Nepal and Sri Lanka. *Caryota urens* is medium sized palm up to 20m tall, bole straight, branched, and elongated inter nodes. It is grown in monsoon climates and peri-humid regions. It prefers moist, shady & cool places, and is slow growing, shade tolerant species.

2.2 Extraction of *Caryota urens* Spadix Fibre

The combination of both mechanical and water retting process was adopted for extraction of fibre from *Caryota urens* Spadix fibre[6]. Fibres found in the inner side were then separated from the outer sheath manually by combing the stems with the serrated needle placed over the wooden board[7]. The separated fibres were washed thoroughly using fresh water and then dried in shade at room temperature (27°C) for 2 – 4 h to ensure the maximum moisture removal[8]. Finally, the fibres were conditioned using the hot air oven at 105°C $\pm$ 2°C until its constant weight is achieved and excess moisture is removed. Without any further treatments the fibres thus obtained were left in their original state for further studies.

2.3 Characterization of *Caryota urens* Spadix Fibre

2.3.1 Chemical Analysis

Cellulose, lignin, hemi cellulose, holo-cellulose and ash content present in *Caryota urens spadix* fibre were measured using solvent analysis and standard test method in terms of weight percentage (wt. %). The Kushner and Hoffer method was used to determine the cellulose content present in *Caryota urens spadix* fibre. According to the Klason method, the lignin content of *Caryota urens spadix* fibre was measured. As per ASTM F 1755-01 standard, *Caryota urens* spadix fibre ash content was measured[9-11]. The holo cellulose of *Caryota urens* spadix fibres was determined according to the method described by Wise *et.al*[12]. The cellulose content and holo cellulose content difference was measured to identify the hemi cellulose content. The average of five samples with standard deviation value has been reported[13].

2.3.2 FTIR Study

Fourier transform infrared spectroscopy of *Caryota urens spadix* fibre was recorded using TENXO 27 in a PR mode with a scan rate of 32 scans min^{-1} and $2cm^{-1}$ resolution in the wavelength range of 500-4000 cm^{-1} at 20°C (room temperature)[14]. The free functional groups present in *Caryota urens* spadix fibres were recorded.

2.3.3 Single Fibre Tensile Test

The tensile strength of *Caryota urens* spadix fibres was measured by a single fibre tensile test method as per ASTM D 3822 standards with a cross-head speed of 0.5mm min^{-1}. Universal tensile testing machine was used with a load cell of 1KN capacity on randomly selected fibre samples (n=20). The entire test was performed with a relative humidity of 65% and room temperature of 21˚C[15, 16].

2.4 Morphological Studies

2.4.1 SEM Analysis

The surface morphology of *Caryota urens* spadix fibre was examined using scanning electron microscope, FFI Quanta 200 to observe structure and surface of fibre. Sample was prepared by coating the fibre with 8 mm gold/palladium using sputter coater apparatus for 90 min to avoid the effects of electron beam charge during examination. An accelerating voltage of 25kV with high energy beam was used to scan the sample with a vacuum level off 1.5×10^{-3} Pa. Then the surface of the sample image was captured with different magnification[14].

2.4.2 X-Ray Diffraction Spectroscopy

The X-ray diffraction spectrometer Shimadzu was used to analyse the amorphous and crystalline phase of the *Caryota urens spadix* fibre with Cu kα (λ=1.5406˚A) as a radiation source at a current of 30 mA and accelerating voltage of 40 kv. In order to obtain an acceptable diffraction pattern all the samples were scanned in 2θ range between 10˚ and 90˚ at a rate of 10˚/min. The crystallinity index of the *Caryota urens spadix* fibre was calculated using the following equation:

$$CI = \frac{I_{min} - I_{max}}{I_{min}} \times 100 \quad \ldots (1)$$

where I_{min} represents minimum intensity of the peak corresponding to crystalline fraction; and

I_{max} represents maximum intensity of the peak corresponding to amorphous fraction. The crystallographic plane was measured using Scherer's formula as shown below:

$$CS = \frac{K\,\lambda}{\beta\, cos\theta} \quad \ldots (2)$$

where K is the Scherer constant (0.94); λ, the X-ray wavelength (0.154nm); β, the peaks at full width half maximum; and θ, the Bragg angle.

2.5 Thermo-gravimetric Analysis

The amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere was measured by thermo gravimetric analysis[10]. The thermal analyzer (model NETZSCH STA 449F3) was used to examine the thermal degradation of *Caryota urens* spadix fibre. The powdered fibre sample was placed on aluminum crucible supported by a precision balance because the instrument consists of a furnace.

The measurement was performed in high purity nitrogen atmosphere at a flow rate of 20mL/min, and thermogram at a heating rate of 30˚C/min records the weight loss in the temperature range RT to 1000°C. To maintain a good contact between sample and thermo-couple, the alumina crucible was used[17].

2.6 Fibre Length and Diameter

Advanced instrument is not possible to use due to brittle nature of the fibre to analyse the length of fibre. Calibrated steel scale is manually used to measure the length of the fibre. The fibre diameter was tested using SEM photography of individual fibre taken along longitudinal direction. Ten different fibre samples were scanned in different longitudinal position to get accurate result[18].

3 Results and Discussion

3.1 FTIR Analysis

The free components as shown in the *Caryota urens* spadix fibre spectrum are observed, within the wavelength range 4000-500cm^{-1}. FTIR spectra show 11 well defined peaks of *Caryota urens* spadix fibre at 3340, 2924, 1733, 1634, 1428, 1371, 1322,1242,1033, 598 and 553cm^{-1} (ref. 19). The peak at 3340 cm^{-1} in the *Caryota urens* spadix fibre indicates the presence of O-H stretching of α cellulose and the peak at 2924 cm^{-1} indicates the presence of C-H stretching of cellulose. The carbonyl peak exists at 1733 cm^{-1} (ref. 20). The peak at 1634 cm^{-1} belongs to carboxyl stretch of C-O, indicating the presence of acetyl group in hemi cellulose [21]. The CH in the plane deformation with aromatic ring stretching peak exists at 1428 cm^{-1}. The peak at 1371 cm^{-1} indicates the presence of aliphatic nitro compound (NO_2 symmetric stretching) of various nitrogen containing compounds. The peak at 1322 cm^{-1} indicates the presence of cellulose absorption at –OH stretching. The C-H bending bond structure of the functional group of alkane's peak exists at 1242 cm^{-1} (ref. 22). The peak at 1033 cm^{-1} indicates the presence of C-O stretching of alcohol (cellulose, hemi cellulose and lignin). The C-O stretching and C-H rocking vibration of cellulose peak exists at 896 cm^{-1}. The peaks at 598 cm^{-1} and 553 cm^{-1} indicate the C-X stretching of organic halogen compounds.

3.2 Chemical Composition of *Caryota urens* Spadix Fibre

The chemical composition of the fibres was strongly influenced by the region, maturity of the plant, extraction condition and methods used to determine the composition[23]. Depending upon the cellulose content in the fibres, their strength and stiffness are concluded. The holo- cellulose and cellulose contents of *Caryota urens* spadix fibre are found to be 79.4 wt.% and 42.1wt. % respectively. The hemi cellulose (30.2 wt. %) content is responsible for the moisture absorption, bio degradation and thermal degradation of the *Caryota urens* spadix fibre[14]. Higher lignin content (21.1wt. %) of the *Caryota urens* spadix fibre keeps water in the fibre and protects against biological attack as well as contributes to the structure, properties and morphology[24]. The increased ash content (4.5 wt. %) decreases the fire resistance characteristics and eliminates the amorphous elements[25].

3.3 Single Fibre Strength

Besides the fibre length and fineness, the fibre strength is considered to be an important fibre property[26]. Fibre strength denotes the maximum tension; the fibre is able to withstand before breaking. It can be expressed as breaking strength and tenacity. The mechanical properties of *Caryota urens* spadix fibre depend largely on the chemical composition, especially the cellulose percentage and cell wall structure[27]. The pre conditioning of fibre is done at 21±1˚C and 65±2% relative humidity. The single fibre is mounted in the jaws of the clamps. All slacks are removed without really stretching the specimen and care is taken to keep the specimen straight within the jaws, ensuring that the fibre sample lays on the line of action between the force and measuring device and the point where the fibre is left from the moving jaw face. Twenty samples are randomly selected for test and a load- elongation graph is plotted to test the fibre samples. The mechanical properties of the *Caryota urens* spadix fibre are determined[14]. The maximum tensile force is found to be 4.37N, young's modulus of *Caryota urens* spadix fibre exhibits 1970-6330 Mpa and the percentage of elongation at break of *Caryota urens* spadix fibre is 3.8% (Fig.1).

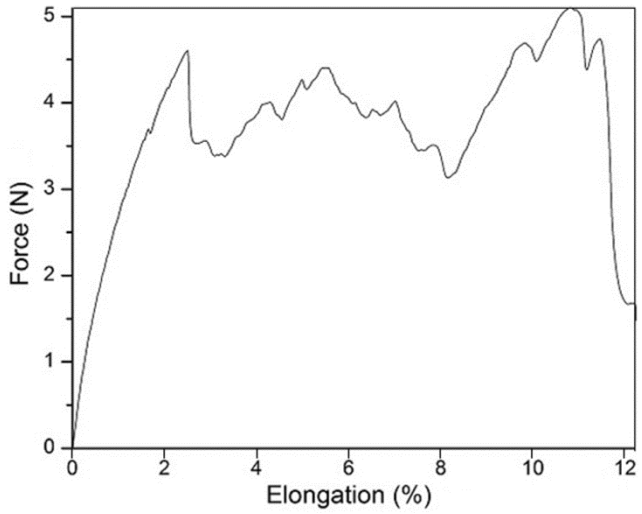

Fig. 1 — Single fibre strength of *Caryota urens* spadix fibre

3.4 SEM Study

The surface morphology of a fibre is a very important factor for determining the ability of fibre to act as a good reinforcement and to resist fibre pull out[28]. To evaluate the *Caryota urens* spadix fibre surface, SEM study was carried out (Fig. 2)[29]. The fibre has a clean and smooth surface and shows a thick layer of uniform deposits over the entire length that is composed of hemicelluloses and lignin [Fig. 2(a)]. It consists of several elementary fibres (fibrils or fibre-cells) joined together in the length direction by pectin and other non-cellulosic compounds [Fig. 2(b)]. This morphology is attributed to the thick outer layers of waxes and other substances (e.g. hemicellulose and lignin), covering the fibre surfaces to protect the cellulose inside [Fig. 2(c)].

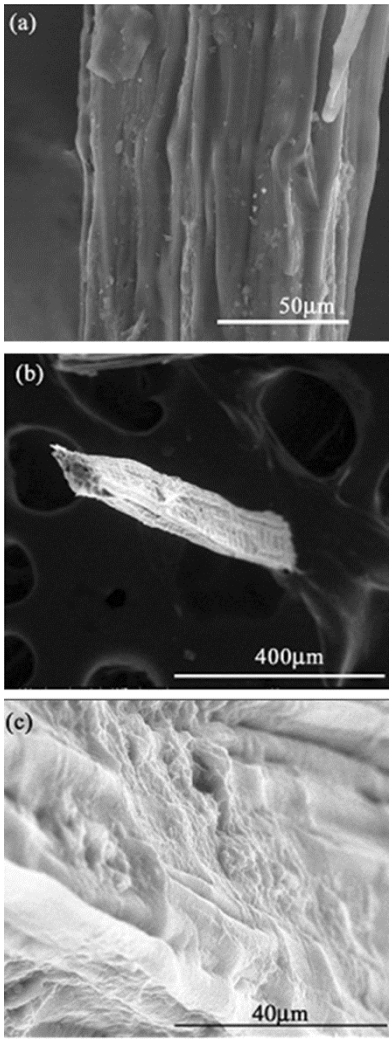

Fig. 2 — Scanning electron microscopic appearance of *Caryota urens* spadix fibre [(a) 50µm, (b) 400 µm, and (c) 40 µm]

3.5 X – Ray Diffraction

Fibre properties are influenced mainly by two important quality, namely crystallnity index, and degree of fibre orientation[30]. Wide angle X-ray diffraction (WAXD) is used to determine the crystal size and crystalline percentage of the sample[31]. The X-ray diffraction spectrum from *Caryota urens* spadix fibre is shown in (Fig. 3). It shows three well defined peaks at 2θ =22.19˚, 2θ =16.60˚ and 2θ =35.32˚. The diffraction peaks indicate that the fibre is semi crystalline. The crystallographic planes (002), (110) and (004), which represent the crystalline peaks of cellulose I, are usually observed in case of natural fibre. The impurities present in the fibre indicate the remaining peaks in the spectrum[14]. The CI value of 90.13% is found higher than hemp (88%), Napier grass (69%) and flax(80%)[32].

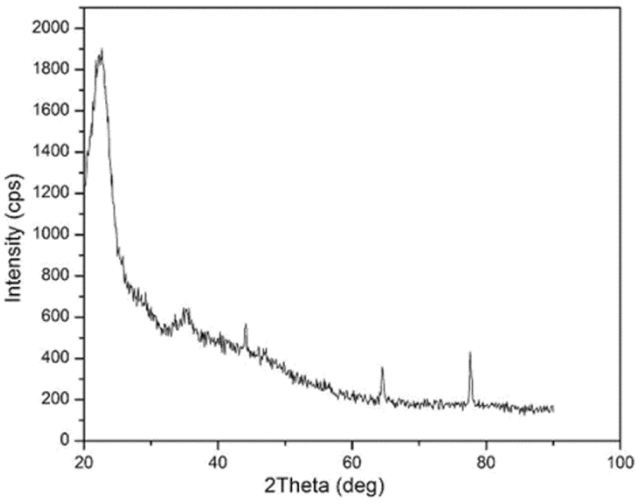

Fig. 3 — X-ray diffraction for *Caryota urens* spadix fibres

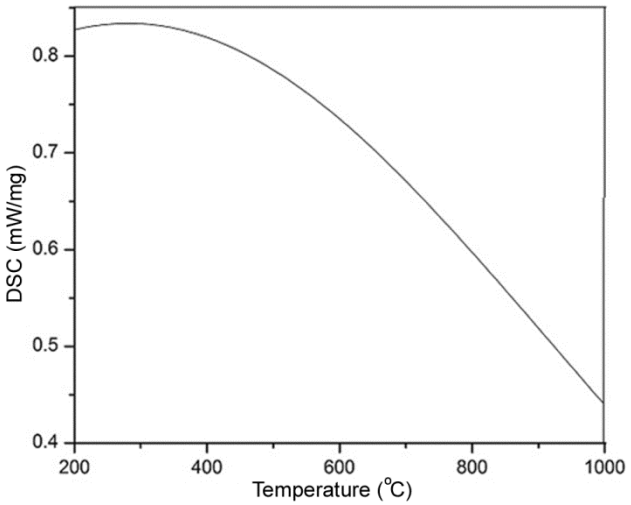

Fig. 4 — Differential scanning calorimeter of *Caryota urens* spadix fibre

The crystalline size is calculated as 3.5 nm for the first crystallographic plane (002) and 0.5 nm for the second crystallographic plane (110). The lower crystal size structure tends to absorb more water than the higher crystal size structure.

3.6 Thermal Analysis

Low thermal stability of natural fibres is used for limiting factors. To study the decomposition pattern and thermal stability of the *Caryota urens* spadix fibres the thermal analysis was performed[33]. DSC shows a board endotherm (peak 400°C) that is extended up to 670.8°C (Fig. 4).

The moisture present in the fibre calculated from this endotherm area is roughly around 50%. DSC does not show any exo or endotherm between 100°C and 220°C, indicating that the fibre doesn't undergo any thermal degradation in this rang of temperature. However, it appears to degrade exothermically beyond 270°C (Fig. 4). The small endotherm observed at around 670°C in the virgin fibre more likely attributes to the melting and volatilization of the fatty and wax materials present in the fibre[34].

3.7 Fibre Length and Diameter

The length of the fibre depends upon the fertility of soil[35]. The length of the *Caryota urens* spadix fibre is found 35.84cm, and its standard deviation is about 2.725 and coefficient of variation is 7.6%. The diameter of fibre observed by SEM is found to be 563.76 μm and CV (23%).

4 Conclusion

The purpose of this study is to investigate the effect of fibre extraction from *Caryota urens* spadix. Fibres are extracted by traditional manual scrabbing process. Physico-chemical and mechanical properties of the sample are studied. Fibre shows the best mechanical properties in terms of strength and rigidity, along with less extensibility. Following inferences are drawn:

4.1 The preliminary analyses of mechanical properties indicate a young's modulus between 1970 Mpa – 6330 Mpa and percentage of elongation at break of 3.8%. The tensile properties of *Caryota urens* spadix fibre confirm that this fibre is one of the best alternative synthetic fibres in composite structure.

4.2 SEM investigation reveals that the *Caryota urens* spadix fibre has a clean and smooth surface and shows a thick layer of uniform deposits over the entire length that is composed of hemicelluloses and lignin.

4.3 The XRD and FTIR analyses show that the fibre are semi-crystalline and shows allotrope from of I_β cellulose. The high percentage of cellulose in *Caryota urens* spadix fibre can offer relatively higher strength and low density of fibre to develop lightweight composite matrix structure. The specific mechanical properties of the *Caryota urens spadix* fibre are higher than those of any other agricultural residues fibre known so far.

4.4 The higher thermal degradation stability of the *Caryota urens spadix* fibre indicates their ability to process with various polymer resins to produce fibre having essential properties. The *Caryota urens* spadix fibre seems to be an effective reinforcement phase in composite for numerous applications.

References

1 Li X, Tabil L G & Panigrani S P E, *J Polym Environ*, 15(1) (2007) 25.
2 Singha A S, Kaith B S & Khanna J, *Bio Resources*, 6(2) (2011) 2101.
3 Suddell B C & Evans W J, *Proceedings, International Symposium on Natural Polymers and Composites* (Embrapa Instrumentacão Agropecuãria), 2002.
4 Gillah P R, Irle M A & Amartey S A, *Annals Forestry*, 6(2) (1998)159.
5 Velasquez J R, Wounaan & Embera, *Econ Bot*, 55(1) (2001) 72.
6 *Annona, the Useful Plants of India* (Publication and Information Directorate, CSIR, New Delhi, India), 1986.
7 Saravanakumar S S, Kumaravel A, Nagarajan T, Sudhakar P & Baskaran R, *Carbohyd Polym*, 92 (2013) 1928.
8 Kommula V P, Oki Reddy K, Shukla M, Marwala T & Varada Rajulu A, *Int J Polym Anal Charact*, 18 (2013) 303.
9 Binoj J S, Edwin Raj, Daniel B S S & Saravanakumar S S, *Int J Polym Anal Charact*, 2(2) (2015) 123.
10 Vignesh V, Balaji A N & Karthikeyan M K V, *Int J Polym Anal Charact*, doi: 10/080/1023666 X 2016. 1175206, (2016)1.

11 Justiz – Smith, Juniorvirgo N G G & Buchanan V E, *Mater Charact*, 59(9) (2008) 1273.
12 Wise L, Murphy K M & Daddiecs A A, *Pap Trade J*, 22(1) (1946) 34.
13 Mayandi K, Rajini N, Ritchipoo P, Sreenivasan V S, Jappes J T W & Alavudeen A, *J Reinf Plast Compos*, 34 (4) (2015) 269.
14 Natarajan T, Kumaravel A & Palanivelu R, *Int J Polym Anal Charact*, 21(6) (2016) 478.
15 Ratna Prasad A V & Mohana Ras K, *Mater Design*, 32 (2011) 4658.
16 Arthanarieswaran V, Kumaravel P A & Saravanakumar S S, *Int J Polym Anal Charact*, 20(4) (2015) 367.
17 Nagarajan K J & Balaji A N, *Int J Polym Anal Charact*. doi: 10.180/1023666x.2016.1160814, (2016).
18 Kanimozhi M, *Int J Sci Res Pub*, 1(1) (2011)1.
19 Samson Rwawiire, George William Luggya & Blanka Tomkova, doi: 10.1155/2013/925198, (2013).
20 Asim M, Abdan Khalim, Jawaid M, Nasin M, Dashtizadeh Zahra, Ishak M R & Enamul Hoque M, *Int J Polym Sci*, 49(7) (2009)1.
21 Mayandi K, Rajini N, Pitchipoo P, Winowlin japes J T & Varada Rajlu A, *Int J Polym Anal Charact*, 21(2) (2016)175.
22 Barbara H Stuart, *Infrared Spectroscopy: Fundamentals and Applications* (John Wiley & Sons), 2004, 82.
23 Keller A, Leupin A M, Mediavilla V & Wintermantel E, *Ind Crops Prod*, 13 (2001) 35.
24 Kalai S, Kaith B S & Kaur I, *Polym Eng Sci*, 49(7) (2009) 1253.
25 Gopinath R, Ganesan K, Saravanakumar S S & Poopathi R, *Int J Polym Anal Charact*, 21(2) (2015) 112.
26 Indran S & Edwin raj R, *Carbohyd Polym*, 117 (2014) 392.
27 Kant Rita & Alagh Preethi, *Int J Sci Res*, 4(7) (2013) 2547.
28 Indran S, Edwin raj R & Sreenivasan V S, *Carbohyd Polym*, 110 (2014) 423.
29 Marcia A S, Spinace, Carlos S lambert, Karen K G & Kermoselli, *Carbohyd Polym*, 77 (2009)47.
30 Manimaran P, Saravanakumar S S, Mithun N K & Senthamarai Kumar P, *Int J Polym Anal Charact*, 12(9) (2016)548.
31 Choudhury A K R, *Textile Preparation and Dyeing* (Science Publisher, USA), 2006.
32 Mohkami M & Talaeipour M, *Bio Res*, 6(2) (2011)1997.
33 Elenga R G, Dirras G F, Gomamaniongui J, Djemia P & Biget M P, *Composites: - Part A*, 40 (2009) 418.
34 Karthik T & Murugan R, *J Fibres Polym*, 14(3) (2013) 465.
35 Subramanian K, Senthilkumar P, Jayal P & Venkatesh N, *Eur Polym J*, (41) (2005) 853.