SYNTHESIS AND CHARACTERISATION OF NANO-SILICA FROM BAMBUSA VULGARIS LEAVES AND A SUITABLE GRAFT POLYOL BINDER FROM RICINUS COMMUNIS OIL.

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ABSTRACT

Bamboo leaves are one among the major agricultural by products and also a potential agro waste which are a rich source of silica. Bio-silica, SiO2 is the most abundant mineral on the earth's crust and basically all photosynthetic plants contain a smaller proportion of it. Bamboo leaves were procured from a local farm and subjected to alkali extraction followed by acid treatment and further calcination to bring out pure nano-silica. The synthesized nano-silica particles were then characterized by UV, FT-IR, XRD, SEM-EDAX analysis and anti-microbial studies and was proven for commercial use. In order to prepare nano-silica polymer composites, there is an ardent need for a binder which was effectively prepared from castor oil which is a natural source of polyol and characterised by UV, FT-IR and H1, C13 NMR to prove its binding properties.

Keywords: Bio-silica, Alkaline extraction, Acid treatment, Calcination, Nano-silica particles, Polyol

1. INTRODUCTION

Bamboo leaves are agro wastes arising after the isolation of stem of the plant Bambusa vulgaris for variety of uses mainly in making furniture. The bamboo leaves ash was investigated to have large silica content of about 75.90- 82.86% 1. Many researches include the production of nano-silica from other agro wastes like rice husk ash2, maize husk3, corn cob4, bagasse5, and bamboo culm6. Bamboo leaves give rise to a serious management problem, due to its large volume at the time of disposal, for which the most common and easy solution is landfilling or incineration in an open field. This is of no use but of a nuisance to the environment, creating massive pollution. This could be overcome by collecting the bamboo leaves and subjecting it to the treatment and production of nano-silica which is of a greater use today. Polymer/silica nanocomposites show better properties when compared to traditional composites due to their nano size7. Nano-silica may be prepared by following a very simple process of alkali treatment followed by acid precipitation at desired concentrations8. Furthermore, there is a requirement for a suitable binder for the process of binding of the nano-silica and the polymer. Thus, an epoxidized, hydroxylated castor oil binder maybe used for the purpose and further characterised.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

Bamboo leaves ash, Sodium hydroxide(3M), Buffer(CH3COONa-CH3COOH), Dilute

 ISSN:1005-748X

hydrochloric acid, castor oil, Con. Sulphuric acid, glycerol, glacial acetic acid, methanol and Double deionized water were used.

2.2 Bamboo Leaves ash preparation

The bamboo leaves were collected from a local farm and were cleaned well and dried for 2 days. Then, the cleaned bamboo leaves were burned in a muffle furnace(model: STXMF112) for 10 hours at 800°C and then left to cool down for a day. The ash obtained was ground into fine powder of uniform size.

2.3 Synthesis of pure silica from bamboo leaves ash

5 grams of the prepared bamboo leaf ash was treated with 3M NaOH solution by weighing exactly 12g of NaOH and making it to 100ml using double deionized water. The solution was covered well with thin foil sheet and heated using a hot plate with magnetic stirrer(model: LMMS-5LC) by constant stirring for 6 hours. It was allowed to settle for a day and the filtrate was isolated. Dilute HCl at 2M concentration was added to the filtrate under constant stirring. The pH was checked using a digital pH sensor(model: SSI-303) and found out to be 2. A buffer solution consisting of sodium acetate and acetic acid was used to maintain the pH level around 8 especially for high purity of the silica. Then the contents were allowed to stand for a day at room temperature. The precipitate obtained was washed several times with water and dried well in a drying oven(model: LDO-A13) for 15 hours to obtain pure nano-silica.

$$Ash + 2NaOH \longrightarrow Na_2SiO_3 + H_2O$$

$$Na_2SiO_3 + 2HCl \longrightarrow SiO_2 + 2NaCl + H_2O$$

Fig. 1. Photograph of burnt bamboo leaves

Fig.2 Photograph of Nano-silica.

2.4 Synthesis of Polyol (Binder)

Epoxidation

30 ml of 30% H_2O2% was inserted into the reactor and 50 ml of glacial CH_3COOH was added. Then 2 ml of 96% H2SO4 was added and stirred at 200 rpm at 40ºC for 1 hour. Then 100 ml of castor oil was added and stirred at 200 rpm at 40 ºC for 3 hours and cooled to room temperature and the oil phase was separated.

 ISSN:1005-748X

Fig.3. Photographs of processing the castor-oil based polyol.

Hydroxylation

100 ml of methanol, 50 ml of glycerol, 2 ml of 96% H2SO4 and 5 ml of deionized water was added into a 350 ml three-necked flask and heated to 40ºC, then the oxidized oil was added and stirred for 2.5 hours at 50ºC and cooled to room temperature. The obtained polyol was further characterized.

3. RESULTS AND DISCUSSION

3.1 Characterization of Nano-silica

UV- Vis Spectral Analysis

The UV- Vis spectral analysis of nano-silica was obtained in the range of 200-800 nm. The UV spectrum was recorded by the UV-1700 series spectrophotometer from Shimadzu, Europe.

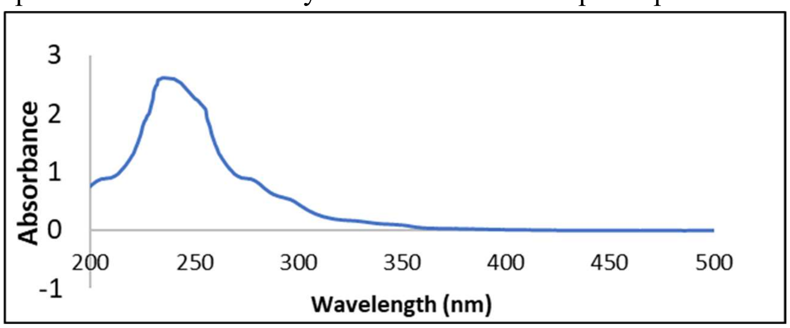

Fig. 4. UV-VIS absorption spectra of the obtained Nano-silica.

The UV-Vis analysis was carried out using 16 ml of TEOS precursor at 650°C and obtained the absorption peak at around 235nm.

FT-IR spectral analysis

The FT-IR spectral analysis was obtained in the range 450-4000 cm-1. The analysis was done by the FTIR- 8400S series spectrometer from Shimadzu, Europe.

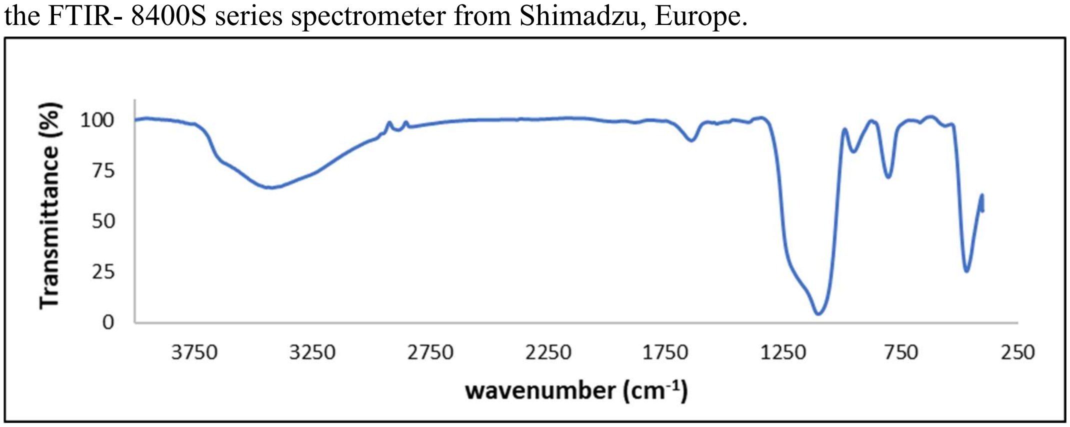

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Fig. 5. Fourier Transform- Infra Red spectral analysis of the prepared Nano-silica.

In the above spectrum, the strong broad band at 3444.63 cm-1 is due to the O-H stretching vibrations owing to the presence of silanol (Si-O—H) by adsorption of a water(H-O-H) molecule. The peak at 2882.42 cm-1 is also due to the O-H stretching due to the intramolecular bond. The bending vibrations of water molecules trapped in the silica matrix produces a peak at 1637.45 cm-1. The Si-O-Si siloxane band stretching accounts for the sharp peak at 1098.39 cm-1. The bending vibration band at 948.91 cm-1, is due to the Si-OH silanol groups. The band at 800.40 cm-1 is due to Si-O symmetric stretching vibrations. The weak peaks at 665.40 cm-1 and 559.32 cm-1 indicates the siloxane stretching vibrations. The sharp band at 467.71 cm-1 is due to the Si-O-Si bending vibrations.

X-Ray Diffraction Analysis

XRD is used to study the crystal structure, composition and physical properties of materials. The XRD analysis of nano-silica below 600°C for 2 hrs showed a broad peak at 2θ= 22.68°. The broad peak confirms the amorphous nature of nano-silica.

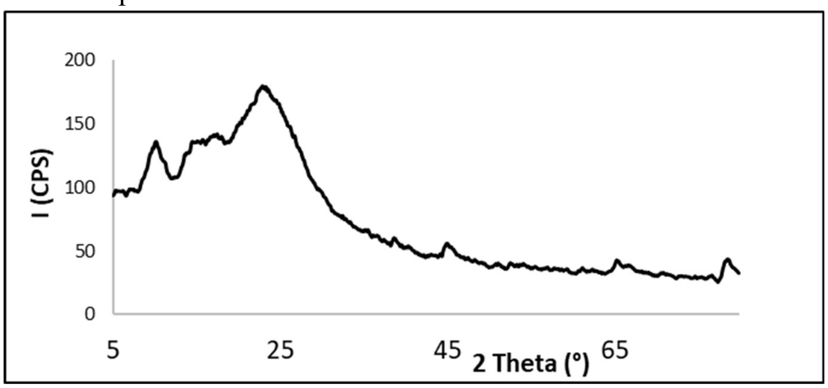

Fig. 6. X-Ray Diffraction pattern of Nano-silica.

Scanning Electron Microscopy (SEM)

SEM provides the microscopic image of the sample by scanning the surface of the sample with a beam of electrons. The SEM images of nano-silica showed the adsorption of spherical shaped silica nano particles there by confirming the amorphous nature of nano-silica.

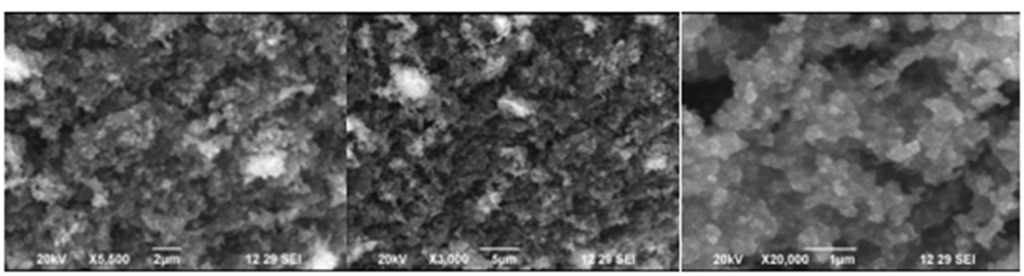

Fig. 7. SEM images of the Nano-silica obtained from the bamboo leaves ash

Energy Dispersive X-Ray Spectroscopy (EDX)

This technique records the x-rays dispersed from the sample by bombarding an electron beam on the sample in order to characterize the elemental composition of the examined volume of the sample. Silicon(Si) and Oxygen(O) are found to be the major constituents of nano-silica.

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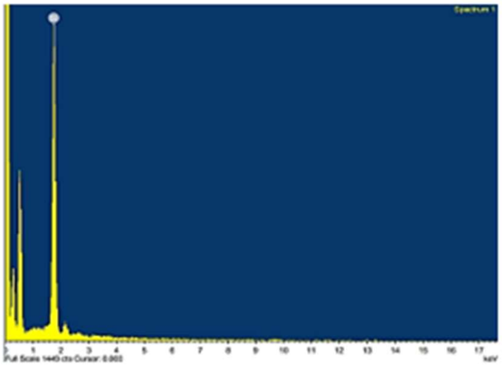

Element	Atomic weight percentage(%)	Weight percentage(%)
Si	31.82	33.63
O	68.18	66.37

Fig. 8. Energy Dispersive X-Ray spectral analysis of Nano-silica.

Anti- Bacterial Activity of Nano- Silica

The nano-silica was subjected to the anti-bacterial study of two positive and two negative bacteriae and the results were interpreted.

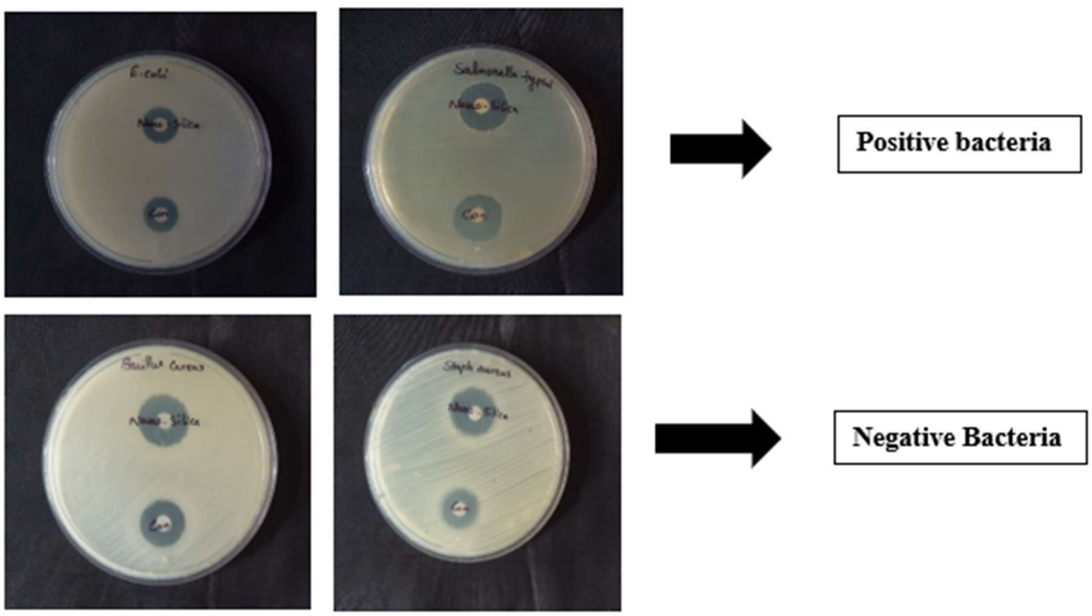

Fig.9. Anti-bacterial activity of nano-silica.

Bacteria	Nano-silica	Control(Amikacin)
Escherichia coli	16 mm	14mm
Salmonella typhi	21 mm	20 mm
Bacillus cereus	20 mm	18 mm
Staph aureus	22 mm	17 mm

Anti-fungal activity of nano-silica

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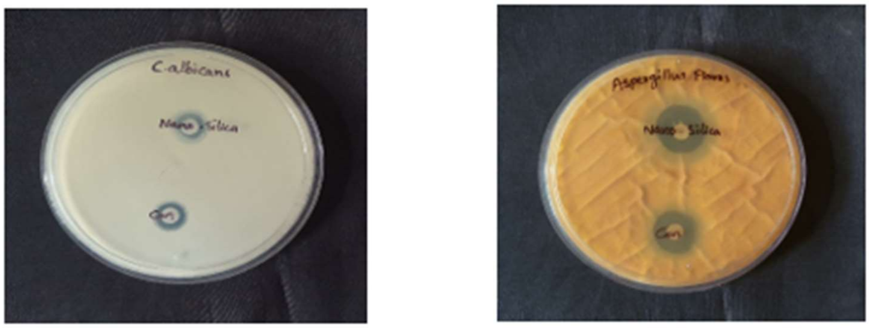

Fig.10. Anti-fungal activity of nano-silica.

The anti- microbial studies of nano-silica have shown best results of anti-microbial activity of nano-silica.

3.2 Characterization of Polyol

H1 NMR of castor oil-based polyol

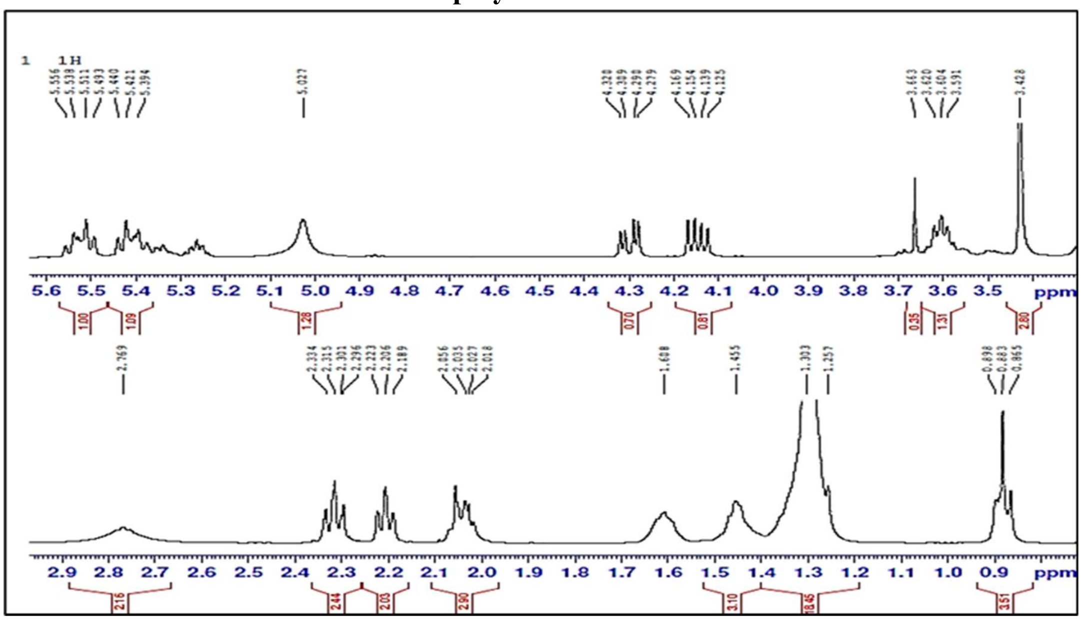

Fig.11. H1 NMR of castor oil-based polyol

The H1 NMR shows that the castor-oil based polyol consists of three hydroxyl groups.

C13 NMR of castor oil-based polyol

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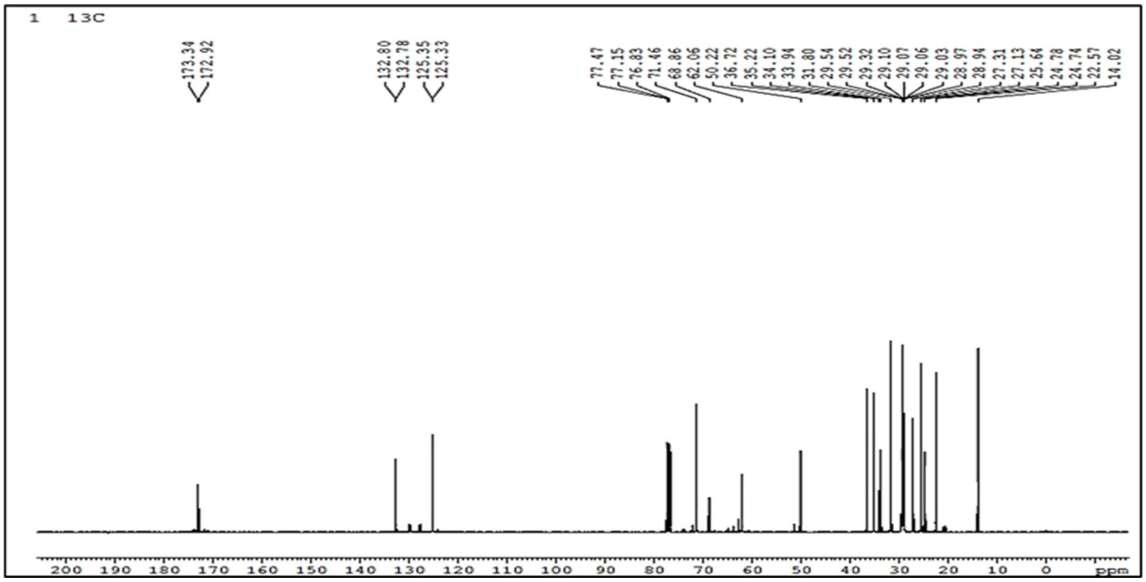

Fig.12. C13 NMR of castor oil-based polyol.

UV Spectra of Polyol

The UV- Vis spectral analysis of the polyol was obtained in the range of 200-800 nm. The UV spectrum was recorded by the UV-1700 series spectrophotometer from Shimadzu, Europe. The UV-Vis analysis was carried out using 16 ml of TEOS precursor at 650°C and obtained the absorption peak at around 336 nm.

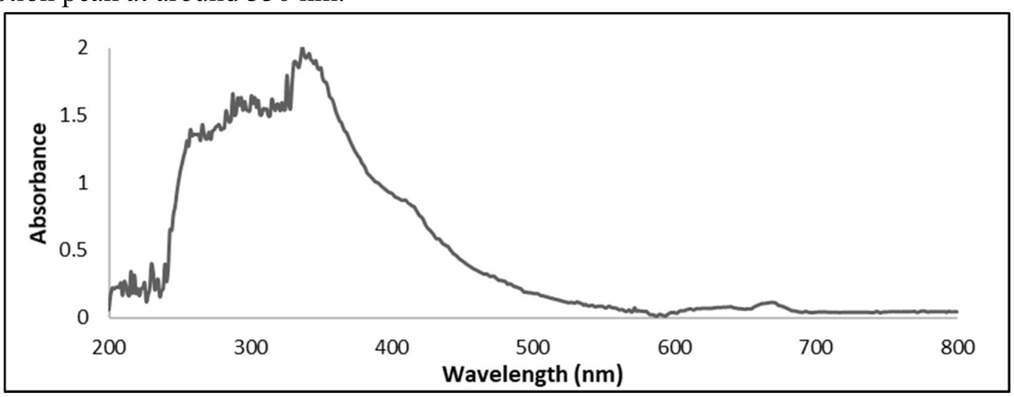

Fig.13. UV absorption spectra of the castor oil polyol.

FT-IR Spectral Analysis

The FT-IR spectral analysis was obtained in the range 450-4000 cm-1. The analysis was done by the FTIR- 8400S series spectrometer from Shimadzu, Europe. The distinct peaks appeared at 3126.39 cm-1, 1744.49 cm-1, 1400.22 cm-1, 1079.09 cm-1, 605.60 cm-1 signify the presence of poly hydroxyl groups in the castor oil-based polyol.

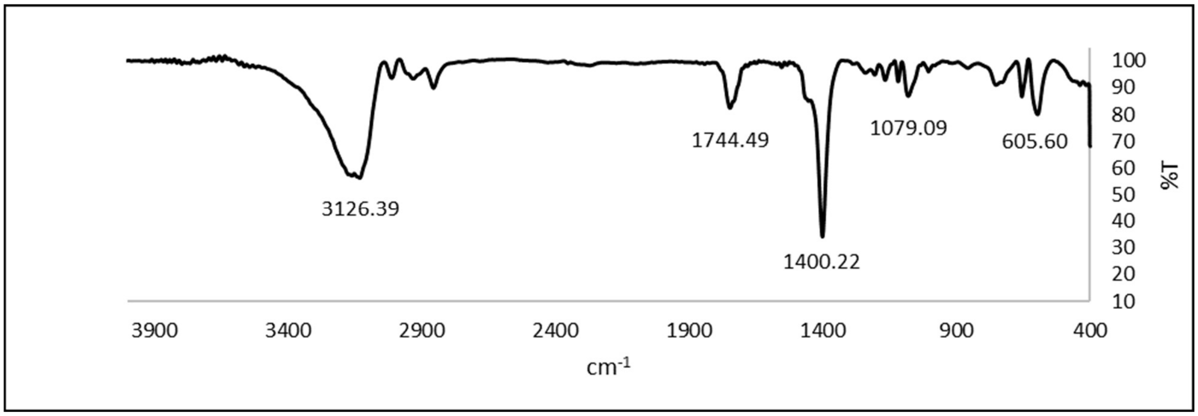

Fig.14. FT-IR Spectra of the polyol.

CONCLUSION

The study encloses the importance of using a bio-degradable bio-waste, namely, bamboo leaves for the synthesis of nano-silica which is confirmed to be amorphous in nature. The materials used in this work are non-toxic by any means to human health and the environment. It also helps with minimizing the waste disposal and management problem. The yield obtained was around 90 (wt/wt)% which is optimal.

DECLARATION OF COMPETING INTEREST

None.

ACKNOWLEDGEMENT

I extend my sincere thanks and regards to ANJAC, Sivakasi and Karunya Institute of Technology and Sciences, Coimbatore.

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