Ultrasonic characterization of compatible chemicals on waste materials for pre synthesis of microwave absorbing material

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Abstract. The efficiency of all microwaves absorbing material (MAM)/radar absorbing material (RAM) is purely depend on its composition particularly organic material like carbon and oxygen. The most of the bio waste are rich in cellulose which contains carbon as main component. The treatment of each bio waste is a vital step before fabrication of biomaterial for different application. The present work describes the ultrasonic analysis of treatment of benzyl chloride with aqueous cellulose. The variation of ultrasonic velocity and other acoustic parameters like compressibility and intermolecular free length with increasing concentration of benzyl chloride suggest possible intermolecular interactions. The presences of such interactions are well explained in terms of morphological changes in waste bio material like rice husk. The presence many reaction sites on the treated rice husk makes suitability for synthesis of biomaterial. The SEM of treated rice husk gives significance explanation for absorption of incident microwave radiation incident on the bio material prepared from this waste.

1. Introduction
Natural fibre/bio wastes are the major component as reinforcement for any advance functional material like bio composites. The compositions of bio waste such as hemicelluloses, cellulose, and lignin generally cover 20–40, 40–60 and 10–25 wt. % for lignocellulosic biomass respectively [1]. The elementary constituents such as carbon, oxygen and other inorganic material are actually deciding factors for fabrication of bio composites. The high percentage of carbon (44.44%) and oxygen (49.39%) in cellulose makes it possible for preparation of activated carbon after proper treatment and surface bleaching of the biomass. The high porosity structure, surface area and low cost of preparation of activated carbon has possesses more attention for fabrication of energy storage device like capacitors, microwave absorbing materials (MAM), stealth material for stealth technology and many more [2]. The poor moisture resistance and low mechanical properties of bio composites limits its application to interior and non-structural design. In composite material the interface between matrix and fibre has a significant contribution to most of the characteristics of composites. The presence of hydrophilic hydroxyl groups at the surface of interface imposes a no. of problem in designing of bio composites. Again the presence of impurity like pectin and waxy substances make the functional group become uncreative. Thus, surface modification is an important step in fabrication of bio material to activate the functional group so that the compositional elements like carbon and oxygen of the cellulosic part of the bio waste or natural fiber become more active. There are many methods and different chemicals are used for surface modification of natural fibers/ biomaterials such as alkali treatment[3],silane treatment[4], peroxide treatment[4], permanganate treatment, isocyanate treatment[5] etc. for activating the functional group as well as to facilitate the fiber materials to...
become active for interaction with incident energy for any allowed energy [6]. After all the treatment of different bleaching agent, it is very necessary that each treatment has some effect on excess use of it on cellulose which reduces the mechanical strength of the fiber. Thus to optimize the use of chemicals for surface modification and to understand the basic mechanism of chemicals with cellulosic materials it is quit interesting to make a systematic study on chemicals by ultrasonic method. Being ultrasonic wave, high frequency can interact at molecular and sub molecular region and correctly estimate the optimized amount of chemicals for the surface modification of bio materials and natural faibers. The present paper describes the ultrasonic analysis of treatment of benzyol chloride with the aqueous cellulose as benzyol chloride. As an efficient bleaching agent benzyol chloride can activate the functional group of the cellulose making hydrophobic nature of the fiber. The interactions of benzyol chloride with the aqueous cellulose are discussed in terms of molecular interaction and then suitable blend are applied to sugarcane bagasse from which the biomaterial can be synthesized for different application like microwave absorption and energy storage devices.

2. Material and Method
The cellulose and benzyol chloride are directly used without further purification as obtained from NICE chemicals, India. The cellulose are dissolved with distilled water for preparation of aqueous solution. The concentrations of aqueous cellulose are sonicated by sonicator of frequency 125 KHz for 15 minute so that the solutions are well dispersed. The bezyol chloride solutions are added with different weight percentage for fixed weight percentage of aqueous cellulose. Similarly, by varying the concentration of aqueous solution four different samples were prepared. All the samples of different weight percentage are sonicated for better dispersion and kept in airtight glass stopper container. The ultrasonic velocities are measured in synthesized solutions using an ultrasonic interferometer working at 2MHZ (Model-M-81S). The velocity measurement was calibrated up to ± 0.01m/s. The densities of pure liquids as well as the mixture were measured with pyknometer within ± 0.0001kg/m3. All the mass measurements were performed by a high precision electronic balance within (±0.001gm). During ultrasonic measurement the temperature of the sample contained within the cell was maintained constant within ± 0.01K by circulating water with the help of a thermostatically regulated temperature water bath through the water jacketed cell. The different acoustic parameters are computed using standard formulas [7].

Isentropic compressibility:

\[ \beta_s = \frac{1}{\rho C^2} \]  

Intermolecular free length:

\[ L_f = k\beta^{1/2} \]  

where “C” is the ultrasonic velocity, “\( \rho \)” is the density of the solutions and \( k \) is temperature dependent constant calculated by [93.875+ (0.375T)] \( \times 10^{-8} \) [8] and \( T \) being the absolute temperature. The residues of sugarcane after extraction of juice were collected and allow to dry 3-4 days in sunlight. The dried parts of sugarcane bagasse are cut in small pieces with length 2-3cm and thickness of 0.5mm. The small pieces of sugarcane bagasse are kept in a beaker containing benzyol chloride and allowed to place in the sonicator for 15-20 minutes. After through ring with distilled water the bagasses are dried at 50°C or 12 hrs. This helps to eliminate the lignin and hemicelluloses residues which results in cellulose degradation [9]. Surface morphology are studied with HITACHI SU 3500 Scanning Electron Microscope for both untreated and treated sugarcane bagasse from which the different information about the sugarcane bagasse can be studied for synthesis of biomaterial.

3. Result and discussions
The acoustic parameters \( \beta \) and \( L_f \) are computed from the measured data of ultrasonic velocity and density of the blended chemicals. The variation of ultrasonic velocity for different concentration of cellulose with increase concentration of benzyol chloride is non linear as shown in figure 1 which clearly indicate the specific interactions between the composition of cellulose and benzyol chloride in presence of ultrasonic wave. The high frequency ultrasonic wave has very small wavelength nearly equal to atomic dimension for which it can influence the atom and subatomic part of the polymer cellulose and gets affected by its different chain network. The initial increase of ultrasonic velocity in low concentration of benzyol chloride indicates that as amount of aqueous cellulose and benzyol
chloride are well dispersed. But the decrease of ultrasonic velocity with increase of benzyol chloride indicates that interaction of oxygen of cellulose and chloride part of the benzyol chloride releases of OH group making the hydrophobic nature.

![Figure 1. Variation of ultrasonic velocity in aq.cellulose/benzyol chloride mixture](image1)

![Figure 2. Variation of compressibility in aq.cellulose/benzyol chloride mixture](image2)

![Figure 3. Variation of ultrasonic velocity in aq.cellulose/benzyol chloride mixture](image3)

Further, with increase of concentration of cellulose amount the ultrasonic velocity decreases due to increase of networking structure of polymer cellulose. As aqueous cellulose are in ionic state, it has greater endurance on benzyol chloride for which the interactions takes place between aqueous cellulose solution and benzyol chloride which makes the solution a poorer solvent medium for the molecules of benzyol chloride. As a result the ultrasonic velocity decreases suddenly with increase concentration of benzyol chloride. The compressibility ($\beta$) increase with increase of concentration of benzyol chloride clearly indicates that there is breakage of bonding of cellulose water which decreases the number of water molecules and decrease in $\beta$ in the higher concentration is due to the increase in electrostriction compression of benzyol chloride around the molecules as shown in figure 2. The effect of ultrasonic wave is also clearly studied in terms of intermolecular free length ($L_f$) which is an important factor for analysis of ultrasonic velocity in treated medium [10]. $L_f$ is the average distance between the surfaces of the two molecules how much they compressed to interact by decreasing the gap between the two atoms as shown in figure 3 clearly supports the variation of compressibility as it is reciprocal of the ultrasonic velocity. The increase in $L_f$ with concentration of aqueous cellulose indicates the significant interaction possible between cellulose and benzyol chloride and suggesting the structural change on addition of benzyol chloride [11-12].
Figure 4. (a) SEM of untreated sugarcane bagasse (b) SEM of treated sugarcane bagasse (c) SEM of treated sugarcane bagasse at 5μm

The effect of ultrasonic treatment on sugarcane bagasse can be verified by SEM. The SEM characterization of untreated and treated sugarcane bagasse shows a clear scientific idea of importance of treatment with benzyl chloride in figure 4(a) (b) and (c). The presence of superficial layer with high percentage of extractives on the surface of fibres are covering the surface which disrupts the strength of reinforcement with matrix as shown in figure 4(a). The treatment of benzyl chloride solution on fiber surface indicates the morphological changes by removing the extractives from the surface of sugarcane bagasse. This removal of extractives results exposure of fibrils by increasing the contact area as shown figure 4(b). In figure 4(c) it is observed that the surface of sugarcane bagasse is clearly forma a net work structure after the treatment of benzyl chloride. This suggest that when this treated fiber mixed with resin material for fabrication of composite this surface structure will enhances the reduction of micro wave intensity by completely absorbing the microwave. The exposure of cellulose component to microwave on the sugarcane bagasse reinforced composites interacting with carbon and oxygen parts of cellulose and increases the dielectric property which will store more energy incident on it.
4. Conclusion
The ultrasonic velocity data and computational acoustic parameters clearly signify the behaviour of
treated medium with the cellulosic part of sugarcane bagasse. The non linear variation of ultrasonic
velocity and acoustic parameters indicates the significant intermolecular interactions such as dipole
dipole, ion-solvent interactions. The formations of H-bond complex formation due to these
interactions are basic factors for the existence of hetero-molecular interaction in treated system in
presence of ultrasonic wave. Thus ultrasonic wave can be considered as a sufficient tool for surface
bleaching and modification of natural fibers for fabrication of composite material. Ultrasonication of
bio waste with suitable modifier provides necessary morphological changes on the fiber which
enhances the better interlocking in reinforcement and matrix of the composite. Thus the ultrasonic
treatment can be suggested for better modification of surface of bio waste or natural fibers by
chemicals of optimum blend as excessive use can destroy the organic component of fibres. This
method may find its application in synthesis of bio composites, designing of different hybrid
composite which has large practical application as dielectric material for greater energy storage
devices and materials.

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