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# Distortion of Ultrasonic Waves in Long – Term Stored Biodiesels and Blends

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***Abstract: Biodiesel is the best alternative fuel to Petroleum Diesel, as it is renewable and environmental friendly. The attenuation of ultrasonic waves of fixed frequency of2 MHz for two commercially available biodiesels, Cotton Seed Oil Methyl Esters (CSOME) and Palm Stearin Methyl Esters (PSME) and their blends with Petroleum Diesel (PD), were studied using an ultrasonic interferometer after long – term storage of two years. Between the two biodiesels, CSOME is rich in unsaturated Fatty Acid Methyl Esters (FAME) and the PSME in saturated FAME. The results showed that there was distortion in the amplitude variation of ultrasonic waves and indicates the presence of insoluble particles due to scattering of ultrasonic waves. The two biodiesels and blends followed a specific trend of amplitude distortion. The ultrasonic absorption estimated was found to be lower than fresh biodiesels and blends except for 30 %, 40 % and 50 % volume blends of PSME biodiesel for which ultrasonic absorption was more than the corresponding fresh blends. The percentage of changes in ultrasonic absorption was in the range 56.92 to 95.86 for stored CSOME blends.For PSME blends the corresponding range was 45.71 to 121.53.The change in ultrasonic absorption for PSME blends was sharp when compared to CSOME blends. The net ultrasonic attenuation is majorly due to scattering. The nature of scattering was different for both the biodiesels and blends that were reflected in amplitude distortions. Ultimately, it was concluded that the ultrasonic attenuation strongly depends on fatty acid profiles of biodiesels.***

***Keywords: CSOME and PSME Biodiesel Blends, Long – Term Storage, Amplitude Distortions, Insoluble Particles, Scattering of Ultrasonic Waves.***

## 1. INTRODUCTION

The entire universe is now looking towards agriculture based renewable and sustainable energy sources such as biodiesel, which is a good replacement for Petroleum Diesel (PD). The advantage of biodiesel is that it is biodegradable and environmental friendly [1, 2]. The biodiesel is miscible with PD in all proportions. The quality levels of biodiesel and blends with PD greatly depend on storage span and temperature. Due to storage the acid value, peroxide value, viscosity will increase, whereas the iodine value will decrease [3]. This is the indication of degradation of biodiesel quality.

Oxidation is the main cause for degradation of biodiesel fuel quality. In general, biodiesel is composed of different Fatty Acid Methyl Esters (FAME). There are two types of FAME, saturated and unsaturated FAME. Unsaturated FAME is more prone to oxidation. The unsaturated double bonds get attack of free radicals and a hydrogen atom get cleaves from the fatty acid chain, then, the oxygen readily reacts with the site and forms hydrogen peroxide.

These peroxides accumulate and then, decompose into aldehydes, alcohols, short chain carboxylic acids and higher molecular weight oligomers. So, oxidation of biodiesel produces insoluble particles [2, 4].

Ultrasonic interferometer is a low cost, sensitive and versatile tool to measure ultrasonic velocity in liquids and their mixtures. It gives very accurate results [5]. The benefit of measurement of ultrasonic velocity is that chemical reactions and physical properties affect the propagation of ultrasound. Determination of ultrasonic velocity gives the information related to physico-chemical and thermodynamic properties of liquid mixtures. It is also useful to study the intermolecular forces [6]. Ultrasonic interferometer is also effectively useful for the determination of ultrasonic absorption in liquids [7].

As a reference, in the blood, the ultrasonic absorption is proportion to weight fraction of protein content. Therefore, it helps in understanding the reaction mechanisms involved in the absorption of ultrasound[8].

In an ultrasonic interferometer, experimental liquid has to collect in to a double wall cell. Inside the cell, there is a source and reflector of ultrasonic waves and the gap between the source and reflector is variable. Between the two standing waves are formed[6]. If the liquid is with suspended particles then they move and coagulate near antinodes. This phenomenon is useful to characterize the suspended particles with in the liquid phase. This phenomenon of coagulation of particles can also be utilized to separate the suspensions from the dispersion liquid medium[9].

Within the interferometer, due to absorption of energy of the amplitude of stationary waves decreases exponentially with increasing source and reflector gap[10].

The amplitude change with respect to distance between source and reflector in an ultrasonic interferometer is given by

$$A = A_o e^{-2\alpha x} \qquad (1)$$

Where, '$A_o$' is initial amplitude and 'A' is the amplitude of ultrasonic wave at a distance 'x' from the source of ultrasound, 'α' is the absorption coefficient. The 'α' is determined for any liquid on determination of amplitude of ultrasound at different positions within the liquid. The estimation of α gives the prediction of nature of elements present in the liquids. From (1), we get

$$Ln\, A = Ln\, A_o - 2\alpha x \qquad (2)$$

It represents a straight line with negative slope of '2α'. So, from the slope α can be estimated [11, 12].

The 'α' is in fact coefficient of attenuation. The attenuation of sound can take place by several mechanisms reflection, refraction, absorption and diffraction[13]. In homogeneous medium attenuation is almost by absorption. In a liquid medium absorption is by thermal conductance and viscosity. For the liquids loss due to thermal conduction is negligible and it is mainly due to viscosity of liquid. So, the absorption coefficient α for the liquids from Stokes formula is:[14].

$$\alpha = \frac{\omega^2}{3\rho v^3}\left\{\left(\frac{4}{3}\right)\eta + \eta^v\right\} \qquad (3)$$

Where, $\omega = 2\pi f$, f is the ultrasonic frequency; ρ is density, v is velocity and η is coefficient of viscosity of liquid. $\eta^v$ is bulk viscosity of liquid.

The 'α' is proportional to square of the ultrasonic frequency. So,

$$\alpha = \alpha_o f^2$$

where, '$\alpha_o$' is constant of proportionality. Therefore

$$\frac{\alpha}{f^2} = constant \qquad (4)\ [8]$$

Ultrasonic scattering occurs in the heterogeneous medium. Scattering generally takes place by the suspended particles in the medium. Due to scattering also, attenuation of ultrasound will take place. Single particle and multiple scattering both contribute to the total scattering.

Therefore, net attenuation is due to absorption and scattering. So, in fact α is coefficient of attenuation. According to Rayleigh, scattering takes place when particle size is less than or equal to wavelength of ultrasound. The Rayleigh's condition for scattering as a function of reflector radius(r) is: [15]

$$\frac{2\pi r}{\lambda} < \frac{1}{10} \qquad (5)$$

Rayleigh's scattering is the case of single particle scattering. This is applicable to low concentration of particles in the liquid. As the concentration of particles increases, the contribution from multiple scattering increases. Along with multiple scattering, the estimation of attenuation due to scattering becomes more complex. The nature of the scattering depends on two factors, frequency of ultrasonic waves and size of the particle scattering ultrasound. As the product of particle size and frequency increases the contributions from single and multiple scattering become dominant than absorption[16].

$$\text{Attenuation coefficient } (\alpha) = \alpha_a + \alpha_s \qquad (6)$$

$$(\alpha_s) = \alpha_{ss} + \alpha_{ms}$$

Where, $\alpha_a$ and $\alpha_s$ are coefficient of absorption and scattering respectively, whereas, $\alpha_{ss}$ and $\alpha_{ms}$ are single and multiple scattering coefficients respectively.

While the ultrasonic waves propagate through the liquid medium with suspended insoluble particles and forms continuous standing waves, the particles transfer towards the antinode zones away from nodal zones where the direction of compressional field within the liquid is from node to antinode that is, maximum at the node and minimum at antinode. This is the single particle interaction in the field. Finally, all the particles settle down in various planes of antinode. Therefore, in the acoustic field scattering of compressional acoustic waves takes place by all the suspended particles. This is the primary interaction between acoustic field and particles. The secondary interaction is particle – particle interaction in the acoustic field where the acoustic field scattered by one particle is again scattered by another particle. For all the particles this is multiplied and leads to multiple scattering[9, 16 – 18].

There are limitations of conventional ultrasonic interferometers for liquids. For homogeneous media in which amplitude of ultrasonic waves change as per (1), the ultrasonic attenuation, which is nothing but absorption can be measured with the help of (2)[11, 12].However, in heterogeneous media with suspended particles where there is possibility for scattering, the scattering coefficient dominates the absorption coefficient. With more concentration of particles, multiple scattering takes place and the problem becomes more complex. Then, the determination of exact scattering coefficient requires the help of advanced set up of interferometer and suitable mathematical model which require the information related to suspended particles such as mass fraction or volume fraction. For some unknown suspension of particles study of this is a difficult task due to lack of this information[17].

Usually, the propagation of ultrasonic waves in a medium is linear. It means that there is a linear relationship between pressure and density variations of the liquid. With the presence of particles across the cross section of plane-acoustic wave the transmission becomes non-linear. Because of the non-linear propagation, there will be distortion in the propagating ultrasonic wave [19].

Any fuel needs storage for its future use. Therefore, storage studies are very important on fuels. As storage stability of biodiesel and blends is low, the concerned studies are very important [20]. The ultrasonic study is one accurate, easy, cost effective and reliable way of doing so [6]. The main intention of the study is in two ways. In one way to study the interaction of ultrasonic waves with stored biodiesels and blends with respect to fatty acid profiles of biodiesels and in another way to identify the quality state of the biodiesels and blends with the help of ultrasonic waves. This kind of information will be useful for the consumer to ensure the purity of biodiesels and blends.

## 2. MATERIALS AND METHODS

The biodiesels procured were Cotton Seed Oil Methyl Esters (CSOME) and Palm Stearin Methyl Esters (PSME), which are commercially available. The CSOME was taken from Southern Online Biotechnologies Pvt Ltd, Hyderabad and PSME from Universal Biofuels Pvt Ltd, Hyderabad, Telangana State, India. PD was collected from an Indian Oil outlet, Hyderabad, India. Five different blends of both the biodiesels in PD were prepared in the volume ratio of 10 %, 20 %, 30 %, 40 % and 50 %.

Then, the biodiesels and blends were stored for a long-term period of two years in a dark cool place at equal levels in semi-transparent plastic bottles as explained earlier [20].This is continuation of the earlier work [20]. After two years, the samples were analysed.

The amplitudes were measured using an ultrasonic interferometer of fixed frequency of 2MHz and variable path length (Make: Mittal Enterprises, Model: F-80, New Delhi, India). In the double walled cell of the interferometer, an ultrasonic reflector was attached with a micrometre screw of least count 0.01mm. The microammeter connected with the interferometer resonates to the positions of nodes on showing passage of maximum current. That way it was possible to identify the positions of nodes and antinodes in the liquid. The amplitudes were noted in terms of current on moving the reflector to different positions separated by five maxima. For every sample three trials were made along the same path. The sensitivity of the ammeter is two micro amperes[12]. The study was carried out at constant room temperature of 28$^{o}$C.

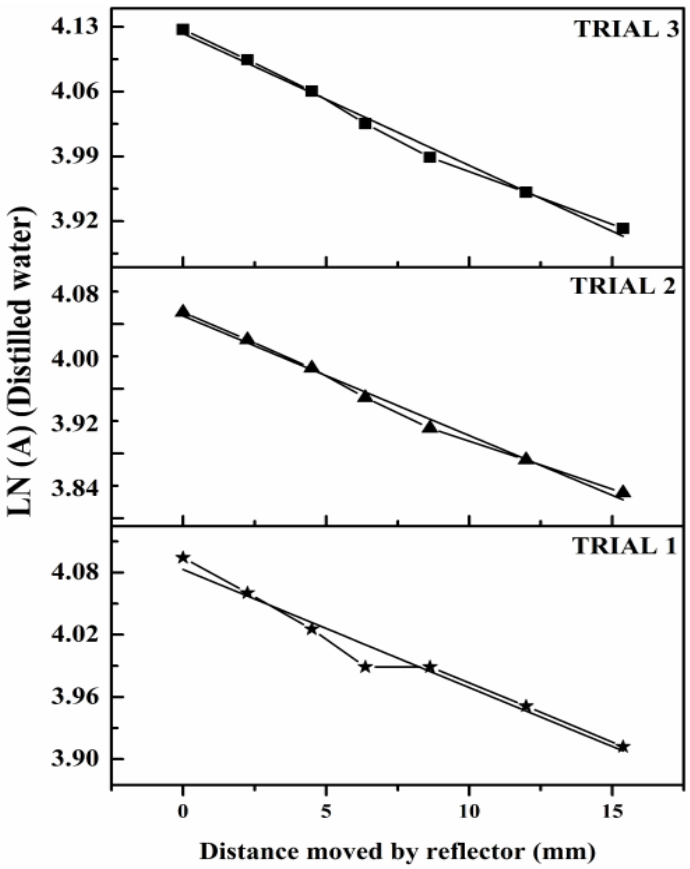


Figure 1 Variation of LN A with respect to distance moved by the reflector for distilled water

For the distilled water, the variation of LN A with respect to the distance moved by the reflector as per (2) is shown in the figure. 1. It was done in three trials. This obtained a very

good straight line as followed by (2) with standard errors 0.00089, 0.00056 and 0.00055, $R^2$ as 0.98. 0.99 and 0.99 respectively for the three trials. This was taken as a reference to analyse the samples.

## 3. RESULTS AND DISCUSSION

The work on characterisation of commercially available CSOME and PSME biodiesels, and their blends with PD has been continuing. Initially, characterization of some key fuel properties of fresh biodiesels and blends were studied. The properties studied were Acid Value, Iodine Value, Peroxide Value and Water Content along with fatty acid profiles of both the biodiesels [21]. Next, ultrasonic absorption in CSOME and PSME biodiesels blends was studied [12]. Later, recently the investigation of molecular interactions on long-term stored CSOME and PSME blends was carried out [20]. All the studies have been carried out with focus on changes with respect to fatty acid profiles of both CSOME and PSME biodiesels. The similar kind of approach was continued in the current study also. Further work is also in progress.

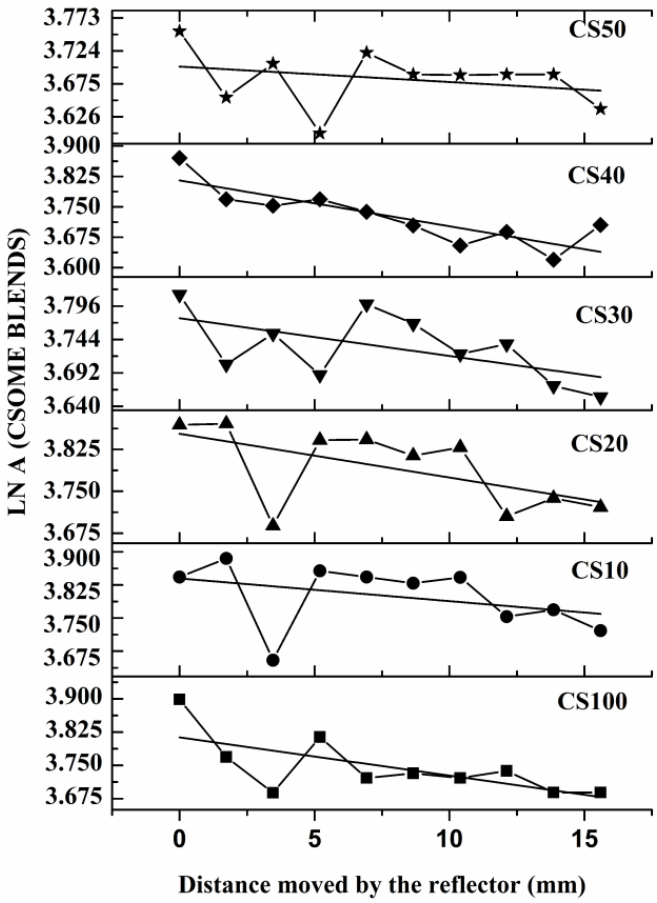


Figure 2 Variation of LN A with respect to distance moved by the reflector for CSOME blends

The variation of LN A with respect to position of the reflector for CSOME biodiesel and blends with PD were shown in the Figure 2 and for PSME biodiesel and blends the variations are in the Figure 3. For every blend, three trials were made and average was recorded. For all the three trials, there was good repeatability of the variation of LN A with respect to position of the reflector. For reference, the variations in three trials for pure CSOME and PSME

biodiesels are shown in the Figure 4 and Figure 5 respectively. These variations are according to (2), which should represent a straight line. However, the solid linear fit lines are well symmetrical to the obtained points. Therefore, it is possible to measure ultrasonic absorption. For the fresh CSOME and PSME blends with PD, the variations were without distortions according to (2) [12]. However, the variations are distortions for both stored biodiesels and blends. These distortions are similar for all the three trials for both the biodiesels and blends. However, nature of the distortions is different for all the blends of both the biodiesels. The distortions in the ultrasonic waves are due to non-linear behaviour of the medium [19].

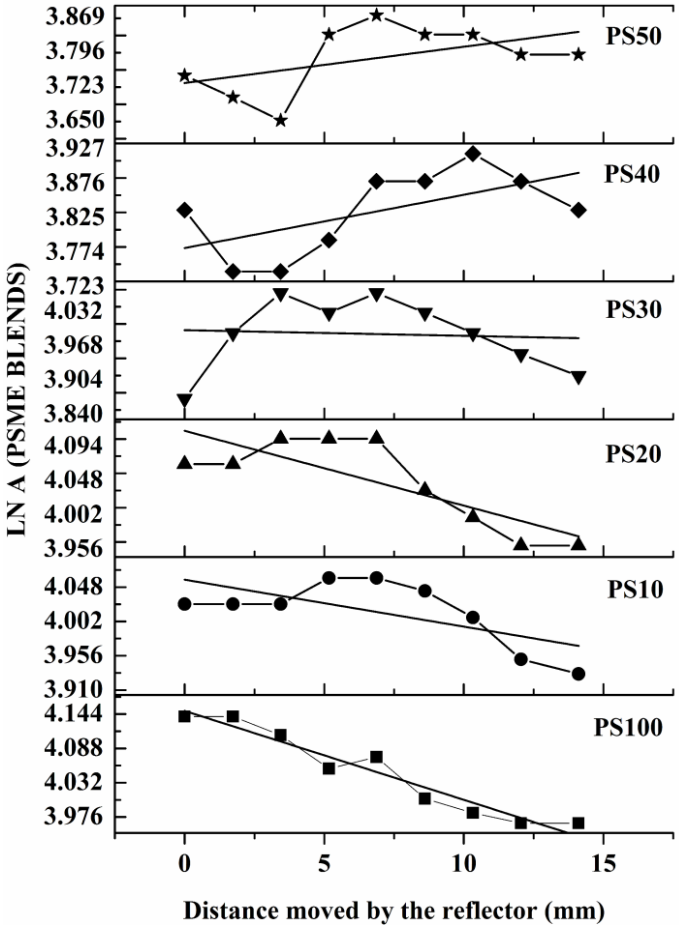


Figure 3 Variation of LN A with respect to distance moved by the reflector for PSME blends

In every trial, the slope of the line changes, but the trend of variation of slope is similar to each other as shown in the Figure 6 and Figure 7. For the convenience and better understanding, the variations of normalised slopes are shown in these figures. For a homogeneous medium, these variations should represent a straight line. With the suspension of the particles in the biodiesel and blends, the medium becomes heterogeneous and scattering takes place[14].Scattering of ultrasonic waves occur based on wavelength and concentration of the suspended insoluble particles[16]. As evident from the settlement of insoluble particles in all the CSOME biodiesel and its blends at the bottom of the containers [20], there is presence of insoluble particles with sufficient concentration and scattering of ultrasonic waves takes place by more domination than ultrasonic absorption. This is evident from the distortion of amplitude of ultrasonic waves in both the stored CSOME and PSME biodiesels and blends with PD. The long-term storage of biodiesels and blends also effects viscosity and related ultrasonic properties as a function of fatty acid profiles of CSOME and PSME biodiesels. This is evident from our recent study [22].

Since the distortions in the biodiesel and blends are because of the scattering by ultrasonic waves, hence the amount of deviation is proportional to scattering. As actual variation of LN A with respect to position of reflector should represent a straight line, it is proper to estimate absorption coefficient ($\alpha_a$), by linear fit of the plots (Figure 2 and Figure 3).The absorption coefficient ($\alpha_a$) of both the biodiesels and blends was estimated and presented in the Table 1. The corresponding variations are shown in the Figure 8 and Figure 9. In both the biodiesel blends, the variation of ultrasonic absorption with respect to blend level is non-linear. However, in the PSME blends there is systematic variation of ultrasonic absorption. Even, the change is rapid for PSME blends. The ultrasonic absorption in all PSME blends is more than CSOME blends. The ultrasonic absorption is positive for 30, 40 and 50 per cent blend volumes of PSME biodiesel. As there is no much difference between the densities of both the biodiesel blends [20], the only reason is the presence of insoluble particles.

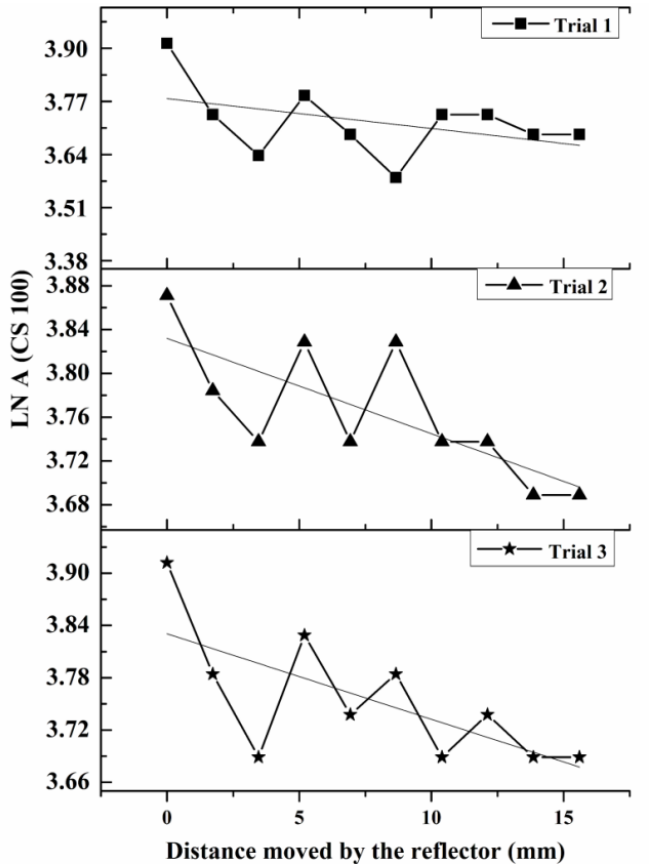


Figure 4 Variation of LN A with respect to distance moved by the reflector for pure CSOME biodiesel

The formation of insoluble particles is due to the oxidation of biodiesels and blends on long – term storage [4]. The sediment formation is there in all CSOME blends. Nevertheless, in the PSME biodiesel and blends there is no formation of sediment at the bottom of the containers [20]. The CSOME biodiesel is rich in unsaturated FAME and PSME in saturated FAME and unsaturated FAME are more prone to oxidation, sediments forms in CSOME blends only [21]. Even though there are amplitude distortions. Therefore, in the PSME biodiesel and blends the insoluble particles are binds to the FAME. Therefore, they did not settle at the bottom of the container.

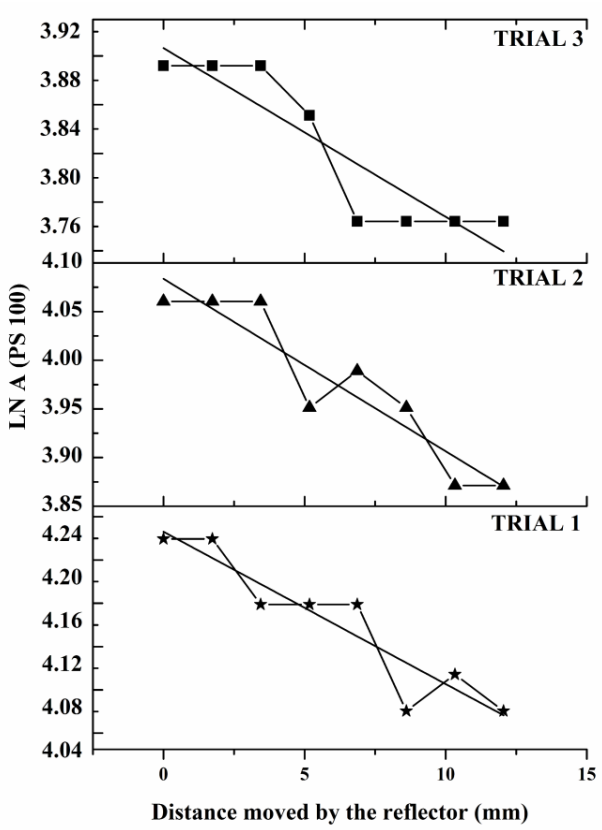


Figure 5Variation of LN A with respect to distance moved by the reflector for pure PSME blends

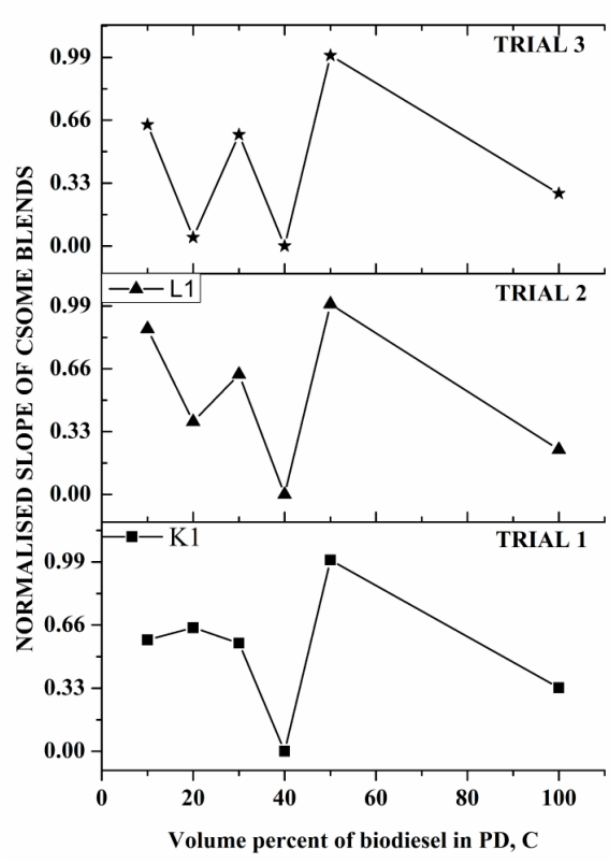


Figure 6 Variation of normalised slope with respect to blend level for CSOME blends

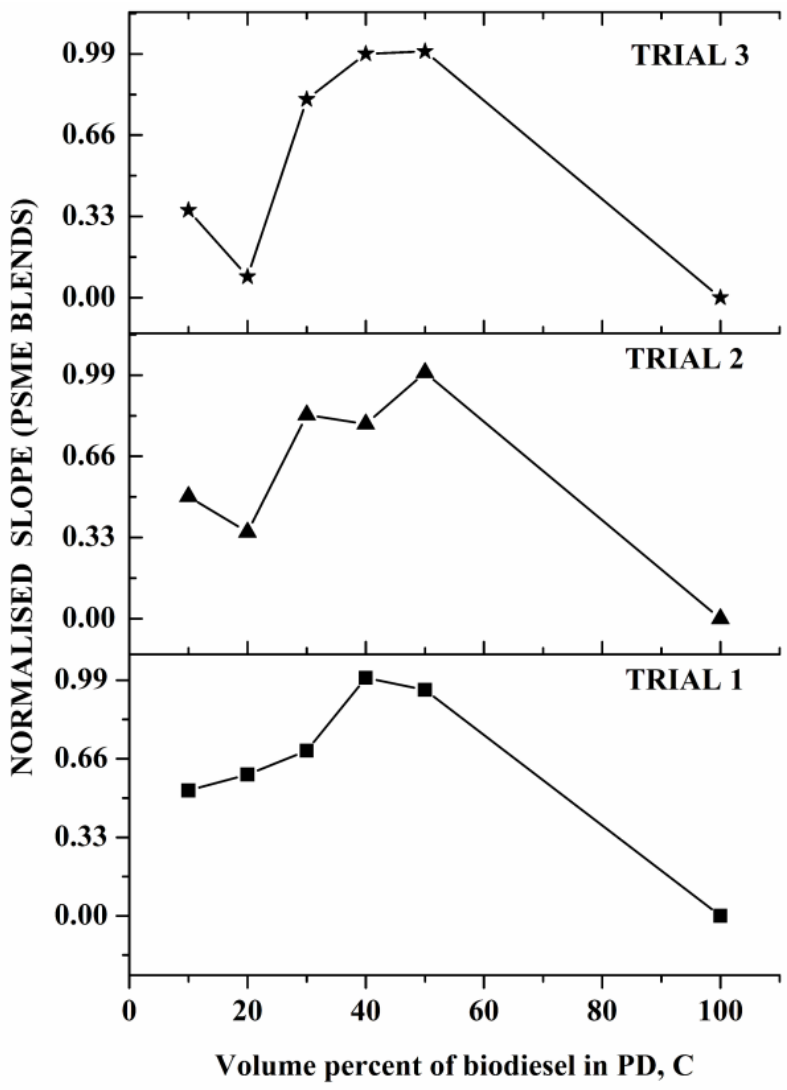


Figure 7 Variation of normalised slope with respect to blend level for PSME blends

Table 1: Ultrasonic absorption of CSOME and PSME biodiesels and blends with PD.

| S No | Volume per cent of biodiesel in PD | Absorption coefficient($\alpha_a$) $mm^{-1}$ | |
|---|---|---|---|
| | | Stored CSOME blends | Stored PSME blends |
| 1 | 10 | -0.0026 | -0.0028 |
| 2 | 20 | -0.004 | -0.0041 |
| 3 | 30 | -0.003 | 0.0011 |
| 4 | 40 | -0.0056 | 0.0021 |
| 5 | 50 | -0.0012 | 0.0028 |
| 6 | 100 | -0.0043 | -0.0076 |

According to the ($\alpha_a$) values of CSOME and blends, the maximum absorption ($0.0056mm^{-1}$) is for 40 % volume blend and minimum for 50 % volume blend ($0.0012mm^{-1}$). For fresh samples, there was maximum absorption again to 50 % volume blend, but minimum was for pure CSOME biodiesel (Table 1, Figure. 8). The percentage of decrease of ultrasonic absorption between fresh and stored CSOME blends are 80, 73.33, 78.57, 56.92, 95.86 and 64.16 respectively for 10, 20, 30, 40 and 50 per cent volume blends.

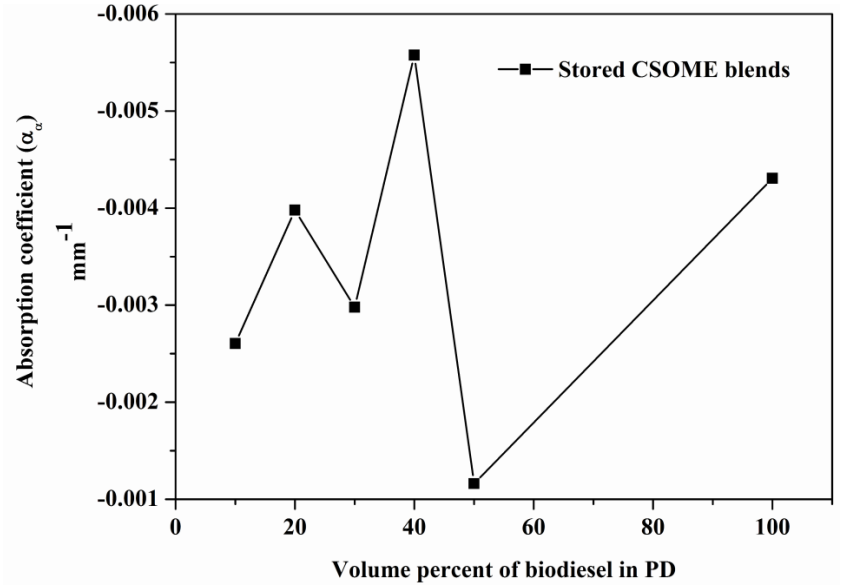


Figure 8 Variation of ultrasonic absorption with respect to blend level for CSOME blends

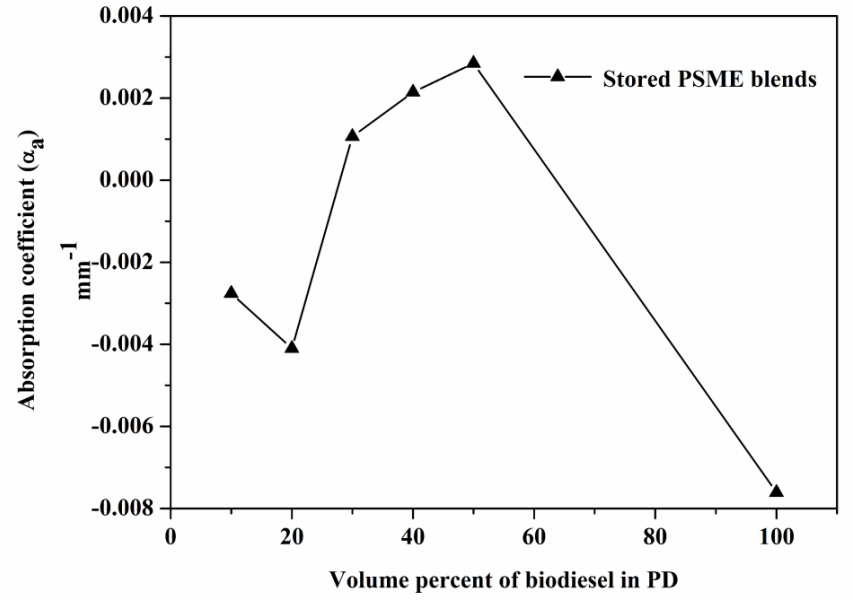


Figure 9Variation of ultrasonic absorption with respect to blend level for PSME blends

For the PSME blends the variation of $\alpha_a$ is much irregular than CSOME blends. Even for 30 %, 40 % and 50 % volume blends the values of $\alpha_a$ came out to be positive instead of negative. This is the indication for very complex multiple scattering by the insoluble suspended particles present. Here minimum absorption is (0.001mm$^{-1}$) for 30 % volume blend and maximum (0.0028mm$^{-1}$) for 50 % blend volume. In contrast to maximum for 30 % volume blend and minimum for 20 and 40 % volume blends. Again, the values obtained are incomparable with that of fresh samples (Table 1, Figure.9).The percentage of decrease between fresh and stored PSME blends are 78.46, 65.83 and 45.71 respectively for 10, 20 and 100 per cent volume blends. Astonishingly the ultrasonic absorption increases by 107.33, 117.5and 121.53 per cent for 30, 40 and 50 per cent volume blends.

The comparison of amplitude distortions between CSOME and PSME biodiesel blends shows different trends of distortions. This is due to the difference in the fatty acid profiles of biodiesels and nature of insoluble particles in biodiesel blends. Specific blends have more

absorption. The trend of distortion has been changing in some regular way with blend level of biodiesel in PD for both the biodiesel blends. The trend of distortion due to scattering is more systematic in CSOME blends in which sediments are formed than in PSME blends, where sediments did not form.

## 4. CONCLUSIONS

From the results, it was found that the presence of suspended insoluble particles created amplitude distortions of ultrasonic waves in both the biodiesels and blends. Depending on amplitude distortions, the presence of insoluble particles was recognised. The amplitude distortions were due to scattering of ultrasonic waves. The trend of distortions noticed as different for CSOME and PSME biodiesel blends. The difference in the trends is attributed to the difference in fatty acid profiles of the CSOME and PSME biodiesels. From the trends of amplitude distortions, in both the biodiesels and blends, it is concluded that the nature of insoluble particles and their distribution is different in both the biodiesels and blends. In the PSME blends, the nature of scattering is more complex than for CSOME blends as there was no formation of sediments. The absorption in stored CSOME biodiesels and blends has been decreased with storage time. Whereas in PSME blends, for specific 30, 40 and 50 per cent volume blends the ultrasonic absorption was increased. By the overall results, it is concluded that ultrasonic absorption and scattering of biodiesels and blends strongly depends on fatty acid profile of biodiesels.

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