

Ultrasonic Studies on Binary Mixture of Methyl Acetate with Cresols

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ABSTRACT: The Ultrasonic velocity (v) studies were carried out at a frequency of 2 MHz (transducer of X-cut quartz crystal) using Ultrasonic Pulse Echo system (model UX-4400-M) on cresols(o-cresol, m-cresol, p-cresol) in Methyl Acetate over entire volume component range at constant temperature of 300.15 K. The values of acoustical relaxation time (τ), attenuation coefficient (α), Absorptivity (A) were computed from the values of Ultrasonic Velocity (v), Density (ρ), and Viscosity (η). It is found that there is a linear correlation between ultrasonic velocity and acidity constant, indicating the dependence of acidity. Correlation of absorptivity(A) and relaxation time (τ) with ultrasonic velocity confirms that the solvent polarity plays an important role in the variation of ultrasonic velocity in the present investigation.

KEYWORDS: Pulse Echo Overlap Technique(PEO), Ultrasonic Velocity (v), Attenuation Coefficient (α), Correlation Absorptivity (A) and Relaxation Time (τ).

I. INTRODUCTION

The study of propagation of Ultrasonic waves in any substance has become an elementary trial to know its properties. Since Ultrasonic study is a useful modus operandi for indulgent in the physicochemical properties of the liquids. It can accelerate reactions that permit the use of unpurified solvents and reagents that can replace phase transfer catalysts which permit the reaction to occur under milder conditions and in some cases even change the reaction pathway. Since ultrasonic waves can radiate through large volumes of liquids, it is well appropriate for industrial applications. For these reasons future applications of ultrasonic in chemical reactions will be diverse[1-4]. When acoustic waves are propagated through a liquid, dissipation the acoustic energy that is associated with it changes the molecular structure of the medium with finite time, which is required for these changes to take place.

In chemical industry, there exists a continuing need for reliable thermodynamic data of binary mixtures. This is particularly true for systems involved in industrial process with their objectives in mind; an attempt has been made to explore the variations of ultrasonic velocity (v), attenuation coefficient (a), density (ρ) and viscosity (η) in Ethyl Acetate and Cresols (o-cresol, m-cresol, p-cresol) as a function of concentration and temperature. These systems have a scope for compellation through hydrogen bonding. Absorption studies may throw more light on the molecular interaction and hence in this study absorption measurements were taken in Ethyl Acetate with different cresols at a frequency of 2MHz and at a temperature of 300.15K[5-8].

Ultrasonic absorption studies in liquid mixtures can help to determine the behaviour in very fast reaction. In many binary liquid mixtures, the characteristic feature is that there exists a prominent maximum in viscosity, ultrasonic velocity and sound absorption at an intermediate concentration which was reported by Gerecze (1977) and Hemmes and Sarvazyan (1980), who studied ultrasonic absorption in diethyl amine and water and explained the excess absorption in this system by assuming a simple two-state model involving equilibrium between hydrogen bonds of like and unlike molecules.

II. RELATED WORK

Esters are chemical compounds consisting of a carbonyl adjacent to an ether linkage. They are derived by reacting an Oxo acid with a hydroxyl compound such as an alcohol or phenol. Esters are usually derived from an inorganic acid or organic acid in which at least one -OH (hydroxyl) group is replaced by an -O-alkyl group. That is, esters are

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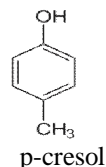
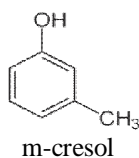
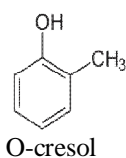
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formed by condensing an acid with an alcohol. Esters are more polar than ethers but less polar than alcohols. They participate in hydrogen bonds as hydrogen-bond acceptors. Because of their lack of hydrogen-bond-donating ability, esters do not self-associate. Consequently esters are more volatile than carboxylic acids of similar molecular weight.

Cresols are organic compounds which are methyl phenols. They are a widely occurring natural and manufactured group of aromatic organic compounds which are categorized as phenols. Cresols constitute one of the most important groups of aromatic organic compounds which occur in three isomeric forms. Like other types of phenols, they are slowly oxidized by long exposure to air and the impurities often give cresols a yellowish to brownish red tint. Cresols have an odor characteristic to that of other simple phenols, reminiscent to some of a "coal tar" smell. They find extensive use in various fields as disinfectants, organic intermediates, textile scouring agents, herbicides, surfactants and in the production of few phenolic resins, tricresol phosphate, salicylaldehyde, coumarin etc. The simplest hydroxyl derivative of benzene is phenol. As structure of phenol involves a benzene ring in its substituted compound the terms ortho(2-methyl phenol), meta(3-methyl phenol), para(4-methyl phenol) are often used in common names. Both ortho and para cresols are used as end products in azodyes. Cresols are less acidic than phenols.

The transmission of ultrasonic waves in a substance has become an essential test to investigate its properties. Ultrasonic velocity measurements have been adequately employed to understand the nature of molecular interactions in pure liquids, binary and the ionic interactions in aqueous electrolytic solutions. In a material scattering and absorption produced by various dynamical interactions which typically arise from anharmonic force between the atoms, the presence of free charge carriers, cycling to localized spin states and the presence of impurities. When acoustic waves are propagated through a liquid, dissipation of acoustic energy that is associated with changes in the molecular structure of the medium results from the finite time, which is required for these changes to take place.



The pulse echo method is much suited to liquids than to gases, mainly because of the much greater absorption of sound by gases, because gases produce much greater damping of the transmitted pulse [9-12]. The first effect means that the reflector must be of longer duration in order to allow for the greater build-up time of the transducer. These two requirements may be conflicting, because of standing waves that are to be avoided, the distance between transducer and reflector must exceed the length of the transmitted train of waves and might thus be such as to give excessive risk of attenuation for satisfactory measurements. The present work has objective to study the behavior of Methyl Acetate and cresols as binary mixtures.

III. EXPERIMENTAL

Technological study of Ultrasonic has been far and wide used for different types of analysis. In Pulse Echo Overlap method a short sinusoidal electrical wave activates the Ultrasonic transducer. The transducer then produces sound wave trains into the liquid inside the cell. This cell is made up of stainless steel which avoids any chemical reactions between the chemicals and the cell. The samples are taken for the present study is of analar reagent grade and procured from Sd. Fine Chemicals Limited, Bombay, India. The liquids are thoroughly distilled to remove the dissolved impurities using standard chemical procedures like steam distillation. Liquids practically are immiscible with water, volatile in steam and possessing a high boiling point. Taking two liquids in separate burettes and with Job's variation method of continuous variation have been used to prepare the mixtures of required proportions.

The ultrasonic velocity measurements are made with the help of microprocessor based ultrasonic Pulse Echo Overlap technique (model UX4400M) with built in time velocity and thickness measuring feature with a frequency of 2MHz. supplied by Roop Telesonic Ultrasonic Ltd, Bombay, India. The internal circuit of (ULTRASONIX4400M) is designed with full solid state version. Further outstanding stability of operation is ensured by dynamic use of integrated circuit and PCB mounted gold switches. It has special memory features which enable one to enter testing procedures like angle

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and type of transducer for permanent storage and direct digital read out of Ultrasonic velocity up to an accuracy of $\pm 1\text{m/s}$.

There are two standard techniques for measuring ultrasonic velocity and attenuation. They are:

- Continuous Wave (CW) method
- Pulse-Echo (PE) method.

The CW method is generally adopted in kHz regions and in low loss specimens. It is possible to achieve high sensitivity. If sample thickness is too small to provide sufficient separation of pulses in PE method, then the CW method is better. Except under such special conditions CW method suffers from:

- ❖ The presence of extra complicating modes of vibrations
- ❖ Boundary effects
- ❖ The need for larger energy dissipations and subsequent heating of the system
- ❖ Lack of good accuracy and precision

The PE method, though generally requiring more complex instrumentation, overcome most of the limitations of the CW method and therefore come into wide spread use. In the commonly used Pulse-Echo (PE) technique, ultrasonic frequency burst is introduced into the sample through a piezo-electric transducer bonded to the specimen with bonding material. The ultrasonic pulse travels through the sample and an echo is registered each time when it returns to the transducer. More often, accuracy and precision of measurement is limited by the above factors rather than instrument. The errors caused by improper selection of the solid sample, bonding material, transducer etc., are the major items. Ultrasonic technique has been widely used for different types of investigations reported by Kalyansundaram. *et al.* (1955). In Pulse Echo Overlap method a shortsinusoidal electrical wave activates the ultrasonic transducer. The transducer then produces sound wave trains into the liquid inside the cell. This cell is made up of stainless steel which avoids any chemical reactions between the chemicals and the cell.

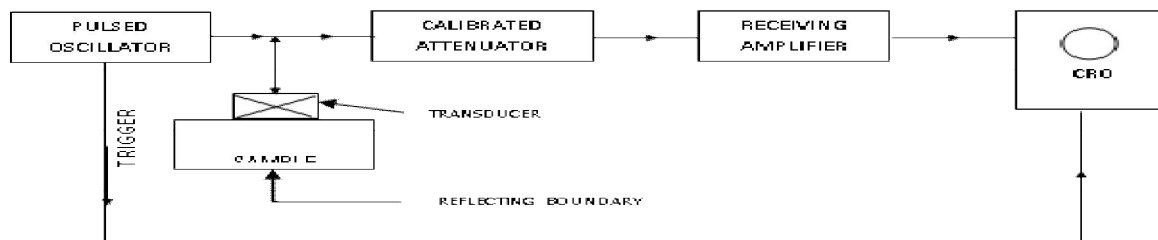


Figure1. Block Diagram of basic Pulse – Echo system

The Pulse Echo Overlap (PEO) method as shown in figure.1 is a very versatile and highly accurate technique for measuring the velocity of the ultrasonic waves in materials of liquids and solids structures. A highly absolute accuracy is obtained from this system, since the method is capable of accurately measuring from any cycle of one echo to the corresponding cycle of the next echo (fig.4). The PEO method is able to handle diffraction (beam spreading) and phase correction properly, so that absolute accuracy of the PEO method may exceed the accuracy of most of the other methods. Ravinder Reddy, B and Linga Reddy, D (1999) improved and used this technique to measure the velocities and attenuations in liquids and solids. The PEO method may operate either with transducer bonded to the specimen or directly with RF bursts. On the negative side, because of transducer effects, the leading cycles of successive echoes are attenuated and trailing cycles are burst up, so that great care has to be taken to obtain proper cycle for cycle match. The phase correction and identification of cycles are to be taken properly which is mentioned in Pulse Superposition technique. In view of the above advantages and accuracy of the system the PEO technique has been adopted for measuring velocity and attenuation.

The binary mixtures of Methyl acetate and phenols were made into three groups on mixing of compound of Esters with O-cresol, m-cresol and p-cresol. The binary liquid is taken into the glass vessel by using 50ml pipette and transducer of 2 MHz frequency is immersed into the liquid. The other end of the transducer is connected to the CRO by BNC cable and Pulse Echo pattern was observed on the screen. The bottom end of the secondary echo pulse of the wave train is coincided to the gate pulse as shown in figure.4. The internal circuit of pulse echo overlap system is

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designed with fully solid state version, which allows immediate calculation of the ultrasonic wave velocity as given in the following equation.

$$v = 2l / t$$

Where l is the liquid length and t is the time interval.

Figure.2. is the Schematic representation of exposure of ultrasound on binary mixture. Ultrasonic velocity and attenuation coefficient are immediately measured with immersion of ultrasonic transducer of 10mm gap between transmitting and reflecting surfaces (in this gap binary mixture is filled) into the same vessel. The other end of the transducer is connected to the ULTRASONIX 4400M (fabricated and supplied by RoopTelsonic Ultrasonix Limited, Mumbai.) by BNC cable and Pulse Echo pattern was observed on the CRO screen [19-20]. This procedure is repeated in all sonication vessels. Figure 3 shows the schematic view of measurement of ultrasonic parameters.

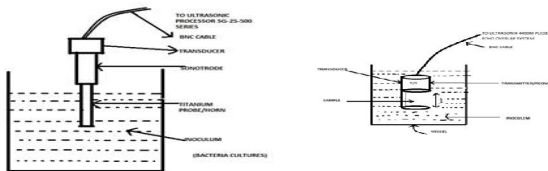


Figure.2. Schematic representation of exposure of ultrasound on binary mixture
Figure 3. Schematic view of measurement of ultrasonic parameters of ultrasound on binary mixture

The densities of all the mixtures have been determined with 10ml. specific gravity bottle and mass (m) of a given volume of the liquid is determined by using a single pan opto-electrical balance. The results of the densities are accurate to $\pm 0.5\%$.

The longitudinal ultrasonic waves are passed through the binary mixture. They are reflected from the reflector face and received by the same transducer, which now acts as a receiver. Attenuation coefficients are used to quantify different media according to how strongly the transmitted ultrasound amplitude decreases as a function of frequency. The transmitted pulse and the received echoes are seen in the CRO screen. Once, the echo pattern is achieved on the screen on the CRO, the pattern is adjusted to a suitable height. The transmitted pulse height is measured in mv/cm and noted as 'a₀' on the screen. The height of first echo is noted as 'a₁' and second echo as 'a₂' and so on. The path length is noted using the micrometer attached to the reflector and is noted as 2l.

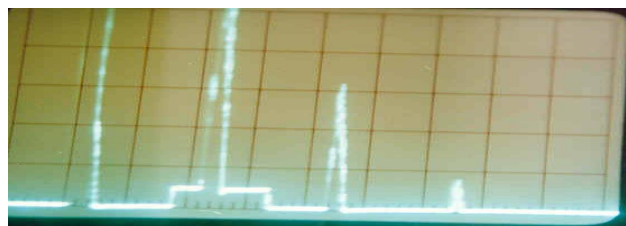


Figure 4. Pulse Echo Pattern on CRO

∴ The attenuation coefficient (α) is calculated as

$$\alpha = 1/2 \ln \log_e (a_0/a_n) \text{ nepers/unit length.}$$

Where a₀ → amplitude of transmitted pulse.

a_n → amplitude of nth echo.

The absorptivity is measured by dividing α by the square of the frequency (2MHZ) of the ultrasonic wave propagated into the liquid media, i.e

$$\text{Absorptivity, } A = \alpha / f^2$$

Where f is the frequency of the ultrasonic wave propagated.

The coefficient of absolute viscosity (η) has been determined using a Cannon- Fenske Viscometer. The results of viscosities are accurate to $\pm 0.3\%$.

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IV. RESULTS AND DISCUSSIONS

As the system is composed of binary components like Methyl Acetate and cresols linearity is observed in ultrasonic velocity and other parameters. Ultrasonic velocity increases with the decrease in volume component of Methyl acetate in the binary mixture. Table1 is the data of ultrasonic velocity, attenuation coefficient, relaxation time, density and viscosity of binary mixture of methyl acetate with o-cresol. Similarly table2 and table3 is the data of ultrasonic velocity, attenuation coefficient, relaxation time, density and viscosity of binary mixture of methyl acetate with m-cresol and p-cresol respectively. Methyl acetate is the basic component here which is more polar. Therefore Ultrasonic velocity decreases with respect to the increase in Methyl acetate concentration. The m-cresol has lower ultrasonic velocity because of high acidity; p-cresol has higher ultrasonic velocity because of the lower acidity. Hence the correlation was done using acidity constant of the 3-isomers. In the substituted phenols the presence of electron withdrawing groups enhance the acidic strength of phenols. This effect is more pronounced when such a group is presenting at ortho and para positions. It is due to the effective delocalization of negative charge in phenoxide ion. This means acidity and electron releasing nature contribute to the variations of ultrasonic velocity of the 3-isomers. The abnormality at the extreme component volume percentage of methyl acetate and m-cresol shows anomalous behavior of higher ultrasonic velocity indicating that the acidity itself is not the controlling factor at these compositions.

The density values increase with respect to the increase in cresol concentration in all the systems. The density values are lesser in Methyl acetate and o-cresol system and larger in Methyl Acetate-m-cresol system. It appears that intermolecular hydrogen bonding between the cresol molecule and cresol solvent molecules may be the factor causing the abnormality.

The attenuation coefficient is a quantity that characterizes how easily a material or medium can be penetrated by a beam of light, sound, particles, or other energy or matter. A large attenuation coefficient means that the beam is quickly "attenuated" (weakened) as it passes through the medium, and a small attenuation coefficient means that the medium is relatively transparent to the beam. The ultrasonic waves are passed through the specimen under study. They are reflected from the opposite face and received by the same transducer, which now acts as a receiver. The Pulse Echo Wave train pattern is observed on the screen. Gate pulse was made to coincide with the bottom of the secondary echo pulse and amplitude is adjusted to 80% of its maximum value. The amplitude pulse noted as a_0 and the amplitude of the n^{th} echo pulse as a_n .

A perusal of ultrasonic velocity (v) in figure1 and attenuation coefficient (α) in figure5 obtained for various binary mixtures of the Methyl Acetate with cresols reveals the following factors.

(i) Attenuation coefficients (α) of binary mixture of Methyl Acetate and p-cresol is lower while compared to binary mixture of other cresols with Methyl Acetate. This means the acidity of cresol is deciding that the attenuation coefficients (α) as observed with ultrasonic velocities (v).

(ii) Attenuation coefficients (α) for o-cresol is higher than other cresols as compared to similar composition of cresols with Methyl Acetate. Generally it can be said that, higher the acidity of the alcohol, the higher will be the attenuation coefficient (α). In case of cresols, with Methyl Acetate binary mixtures figure1, it is observed that p-cresol > m-cresol > o-cresol.

Figure2 and figure3 show the variation of density and viscosity of cresols and is in good agreement with the theory postulated by Rajagopal et al [4]. The relaxation time shown in figure4 and absorptivity shown in figure6 shows the decreasing trend with increasing component volume over entire range of methyl acetate in cresols. This result indicates weak molecular interaction between methyl acetate and cresols.

TABLE1. Ultrasonic velocity (v), density (ρ), viscosity (η), Relaxation Time (τ), Attenuation Coefficient (α), Absorptivity (A) of Binary mixture of Methyl Acetate in O-cresol at 300.15 K

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| Methyl Acetate | O-Cresol | Ultrasonic Velocity (m/s) | Density (kg/m ³) | Viscosity (poise) | Relaxation Time x10 ⁻⁷ sec | Attenuation Coefficient (α)x10 ³ | Absorptivity (A)x10 ⁻⁸ |
|----------------|----------|---------------------------|------------------------------|-------------------|---------------------------------------|---|-----------------------------------|
| 90 | 10 | 1105 | 0.8945 | 0.595 | 7.8477 | 48.0146 | 1.2003 |
| 80 | 20 | 1121 | 0.9082 | 0.680 | 7.70715 | 47.6783 | 1.1919 |
| 70 | 30 | 1151 | 0.9219 | 0.782 | 8.1088 | 48.1083 | 1.2027 |
| 60 | 40 | 1213 | 0.9356 | 0.807 | 8.6734 | 49.7937 | 1.2448 |
| 50 | 50 | 1231 | 0.9493 | 0.892 | 9.8842 | 51.5036 | 1.2875 |
| 40 | 60 | 1243 | 0.9630 | 1.021 | 11.6004 | 53.6649 | 1.3416 |
| 30 | 70 | 1225 | 0.9767 | 1.275 | 14.3969 | 58.8061 | 1.4701 |
| 20 | 80 | 1319 | 0.9904 | 1.615 | 14.6767 | 66.8993 | 1.6724 |
| 10 | 90 | 1350 | 0.9624 | 2.125 | 18.8786 | 79.4849 | 1.9871 |

TABLE 2. Ultrasonic velocity (v), density (ρ), viscosity(η), Relaxation Time(τ), Attenuation Coefficient(α), Absorptivity(A) of Binary mixture of Methyl Acetate in m-cresol at 300.15 K

| Methyl Acetate | m-Cresol | Ultrasonic Velocity (m/s) | Density (kg/m ³) | Viscosity (poise) | Relaxation Time(τ) x10 ⁻⁷ sec | Attenuation Coefficient (α)x10 ³ | Absorptivity (A)x10 ⁻⁸ |
|----------------|----------|---------------------------|------------------------------|-------------------|--|---|-----------------------------------|
| 90 | 10 | 1119 | 0.8954 | 0.552 | 6.5710 | 46.3611 | 1.1590 |
| 80 | 20 | 1147 | 0.9101 | 0.595 | 6.0838 | 40.1267 | 1.0031 |
| 70 | 30 | 1180 | 0.9247 | 0.681 | 7.0417 | 47.1143 | 1.1778 |
| 60 | 40 | 1232 | 0.9335 | 0.850 | 7.9981 | 51.2587 | 1.2814 |
| 50 | 50 | 1270 | 0.9540 | 1.021 | 8.8384 | 54.9452 | 1.3736 |
| 40 | 60 | 1295 | 0.9599 | 1.105 | 9.1524 | 55.7972 | 1.3949 |
| 30 | 70 | 1335 | 0.9833 | 1.530 | 11.6407 | 68.8412 | 1.7210 |
| 20 | 80 | 1376 | 0.9904 | 2.041 | 14.5051 | 83.2252 | 2.0806 |
| 10 | 90 | 1416 | 1.0126 | 2.975 | 19.537 | 108.9301 | 2.7232 |

TABLE 3. Ultrasonic velocity (v), density (ρ), viscosity(η), Relaxation Time(τ), Attenuation Coefficient(α), Absorptivity(A) of Binary mixture of Methyl Acetate in p-cresol at 300.15 K

| Methyl Acetate | p-Cresol | Ultrasonic Velocity (m/s) | Density (kg/m ³) | Viscosity (poise) | Relaxation Time(τ) x10 ⁻⁷ sec | Attenuation Coefficient(α) x10 ³ | Absorptivity(A) x10 ⁻⁸ |
|----------------|----------|---------------------------|------------------------------|-------------------|--|---|-----------------------------------|
| 90 | 10 | 1131 | 0.8942 | 0.553 | 6.4462 | 44.9984 | 1.1249 |
| 80 | 20 | 1152 | 0.9077 | 0.595 | 6.5857 | 45.1342 | 1.1283 |
| 70 | 30 | 1172 | 0.9212 | 0.683 | 6.7228 | 45.2873 | 1.1321 |
| 60 | 40 | 1204 | 0.9348 | 0.765 | 9.1978 | 60.3132 | 1.5078 |
| 50 | 50 | 1243 | 0.9481 | 0.865 | 7.755 | 49.2564 | 1.2314 |
| 40 | 60 | 1274 | 0.9616 | 1.105 | 9.4392 | 56.3752 | 1.4093 |
| 30 | 70 | 1322 | 0.9751 | 1.445 | 11.3056 | 67.5172 | 1.6879 |
| 20 | 80 | 1373 | 0.9980 | 1.995 | 14.1387 | 81.3002 | 2.0325 |
| 10 | 90 | 1430 | 1.0021 | 3.570 | 23.2281 | 128.244 | 3.2061 |

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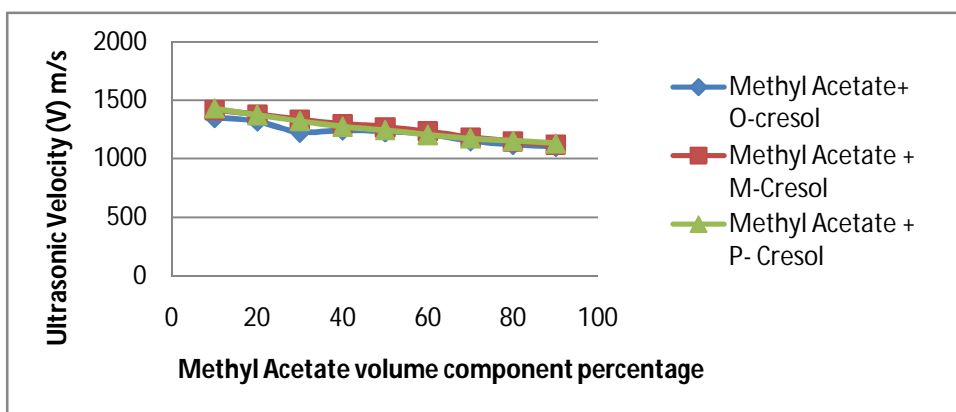


Figure1. Comparison velocities of o-cresol, m-cresol and p-cresol with methyl acetate

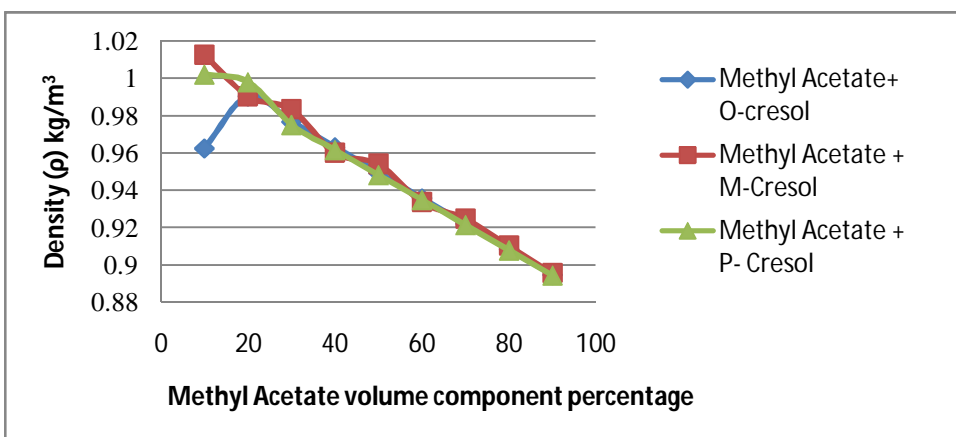


Figure2. Comparison Densities of o-cresol, m-cresol and p-cresol with methyl acetate

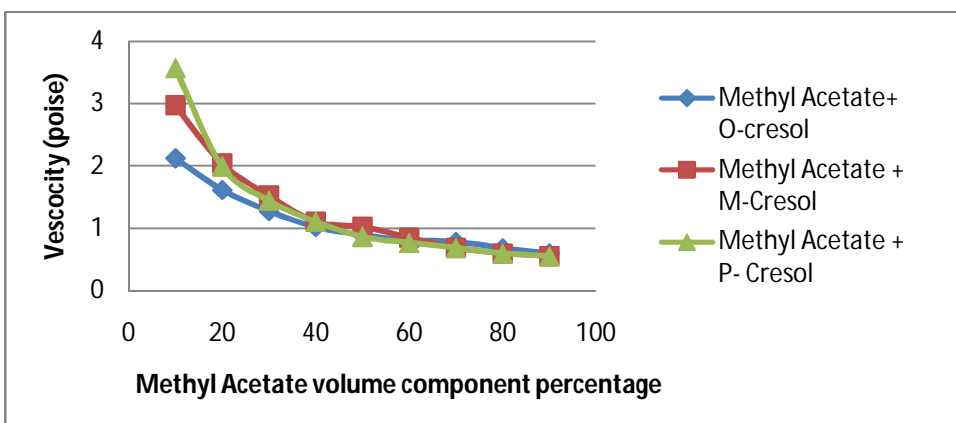


Figure3. Comparison viscosities of o-cresol, m-cresol, and p-cresol with methyl acetate

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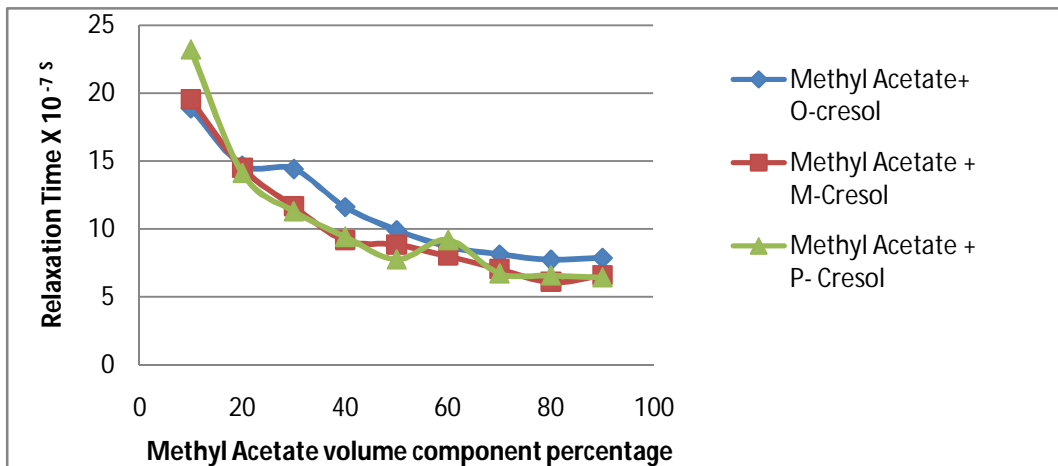


Figure4. Comparison Relaxation time of o-cresol, m-cresol, and p-cresol with methyl acetate

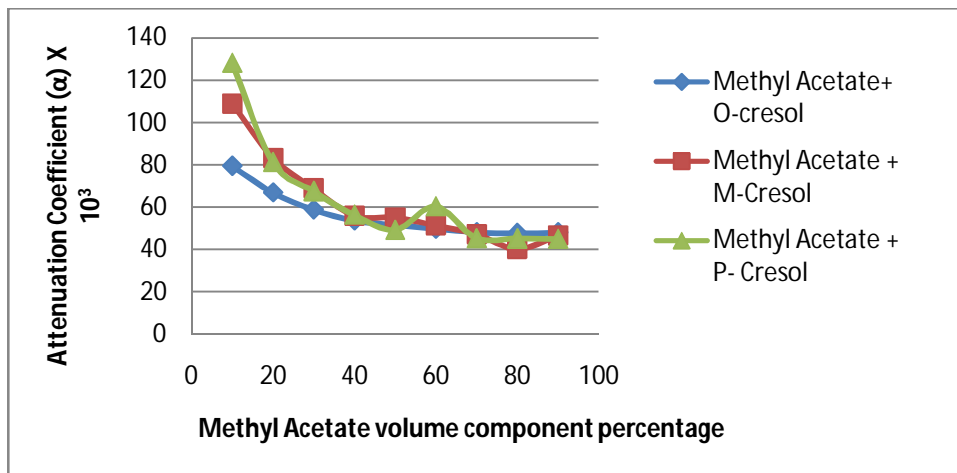


Figure5. Comparison attenuation coefficient of o-cresol, m-cresol, p-cresol with methyl acetate

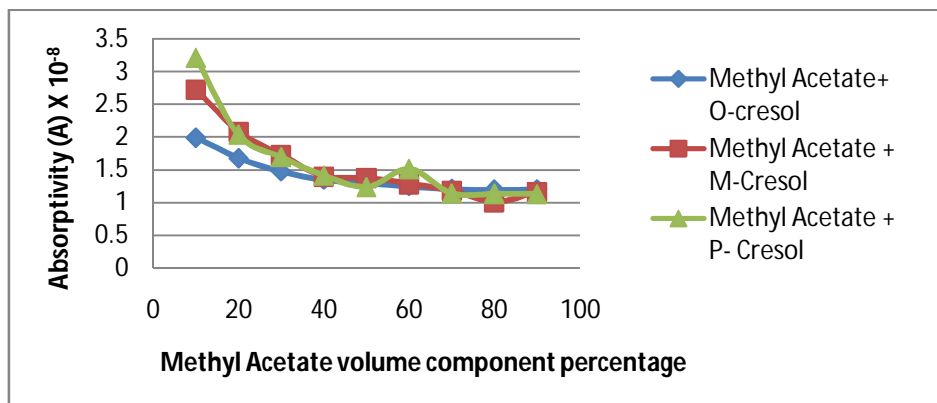


Figure6. Comparison absorptivity of o-cresol, m-cresol, p-cresol with methyl acetate

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V. CONCLUSION

Ultrasonic Investigation studies are carried out on o-cresol, m-cresol and p-cresol with Methyl Acetate over entire mole fraction range of 0-100%. It is found that ultrasonic velocities increase with the chain length of cresols. In case of cresols, with Methyl Acetate binary mixtures it is observed that p-cresol > m-cresol > o-cresol. Results are in good agriment with S.V. Ranganayakul et al (2003).

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