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Synthesis and Characterization of Visco-Elastic (VE) Polyurethane Foam

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ABSTRACT: One of the important applications of polyurethanes is polyurethane foam. Flexible type of polyurethane foam is divided into many types and one of them is visco-elastic foam. Visco-elastic foam is unique foam showing both viscous and elastic behavior at same time. Visco-elastic foam covers major applications of flexible polyurethane foam. Visco-elastic foam samples were synthesized on lab scale by cup foaming. All samples were characterized by finding density of each sample. Cream time, gel time and rise time were also estimated for each sample. The chemical compositions of all polyurethane foam samples were characterized by FTIR analysis.

KEYWORDS: Polyurethanes, Visco-elastic Polyurethane foam, Cup Foaming, FTIR.

1 INTRODUCTION

Polyurethanes are broad class of materials used widely in many applications. Polyurethanes are also written as PUR. Polyurethanes also called as urethanes are characterized by urethane linkage -NH- C (=O) - O - . This linkage is formed due to the reaction of isocyanate group with hydroxyl group [1-3].

The total annual production of urethanes in the world is estimated to be 8 million tons per year and is increasing 4-5 % yearly. Polyurethane foams can be classified in to the two major groups depending on their physical properties. Flexible and rigid foams are low density foams and semi rigid foams have medium density [4-8].

The structure of polyurethane foam is formed due to the foaming system in which all the constituents are mixed and they rise to form an elongated structure having closed or open cells [9-11]. Polymeric (Visco-elastic) foams are used in many applications due to their low weight, and sound- and shock-absorbing properties. Polyurethane foam is a complex engineering material. Material Properties also depend on temperature and humidity and one of the important behaviors of foaming products is visco-elastic behavior [12-14].

Visco-elastic foam technology lead to the new innovation in the field of flexible polyurethane foams. Due to unique characteristics of visco-elastic VE foam it has captured the major applications of flexible polyurethane foam. Visco-elastic memory foam was first developed by NASA in 1970s to help relieve the astronauts from intense lift off. NASA developed a pressure relieving foam that was temperature sensitive and it evenly distributed body weight. NASA released this material in public in 1980s and originally it is proved unstable for commercial use then after a long research and development on visco-elastic VE foam by NASA they succeeded in developing a proper formulation and proper process for manufacture of visco-elastic foam that is commercially a viable product. Visco- elastic foam is also called memory foam and it is due to the reason that it remembers the shape of the object which is placed on it and when the object is removed from it the shape mark of the object is placed on the foam surface [15-18].

2 MATERIALS & METHOD

2.1 MATERIALS

Chemicals used for the experimentation include Modified MDI (Methylene diphenyl di-isocyanate) which is the blend of toluene diisocyantae (TDI) and MDI, Polyether Polyol, additive (Benzyl Alcohol), filler ($CaCO_3$), catalyst (Stannous octoate). All chemicals are of commercial grade and are used without any further treatment. Calcium carbonate would be used as filler for density load build and residue reduction [15]. Instead of using conventional polyether polyol now different new types of polyols can also be used in synthesis of polyurethane foam like soya bean polyol, palm oil polyol, and sugar cane bagasse polyol [19-21].

2.2 METHOD

Visco-elastic foam was prepared using a standard hand mix procedure or cup foaming. Equipment used was Electric Mixer, Paper/ Plastic cups, Stirrer, Stop watch and Weight Balance. Foaming systems and environment was kept at constant temperature. Measured quantity of polyol was weighed in a plastic or paper cup by using electronic weight balance. Catalyst, water, blowing agent and surfactant (if used) were added in it with gentle stirring with spatula. Polyisocyanate component was weighed in a separate cup. Polyisocyanate was poured into the cup containing polyol and was thoroughly mixed for five seconds. Foaming mixture was allowed to expand and rise in the same cup. Foam block was cured at room temperature overnight. Foam samples were cut for testing from upper parts of the foam cured. Different batches were prepared by changing the quantity of chemicals used and also by changing the type of chemicals. One industrial sample of visco-elastic foam was also taken to compare results of lab scale synthesized foam with it. Five samples of visco-elasatic foam were synthesized with different type of chemicals and by varying the quantities of chemicals used. Complete formulation details for synthesis of visco-elastic foam with varying the percentages and type of chemicals are indicated in tabulated form in Table 1.

Table 1. Experimental data for synthesis of visco-elastic foam

Batch No.	**Modified MDI (Blend of TDI & MDI)**	**Polyether Polyol**	**Water (Drops)**	**Additive (Benzyl Alcohol)**	**Filler ($CaCO_3$)**	**Catalyst (Stannous octoate)**	**Results**
1	7 gram	12 gram	3-4	Nil	Nil	Nil	Soft VE foam
2	7 gram	12 gram	Nil	2-3 drops	Nil	Nil	Soft and fine VE foam
3	7 gram	12 gram	Nil	Nil	0.15 gram	Nil	VE foam with rigid surface
4	7 gram	12 gram	2-3	2 drops	Nil	Nil	Soft and fine VE foam
5	7 gram	12 gram	2-3	2 drops	Nil	0.1 gram	VE foam with rapid rise

3 CHARACTERIZATION

3.1 CALCULATION OF DENSITY

Density is a structural property and it is defined as mass per unit volume. Different physical properties of foamed polyurethanes are found through apparent density. For simple shaped samples density can be calculated by weighing it and then by finding its volume through linear dimensions but in the case of complex shaped materials especially polyurethane foams and elastomers the method used for calculating density is described in DIN 53479.

In this method the sample is first weighed when it is dry then it is again weighed after dipping it into the test liquid. In this case test liquid taken is water. Density can be calculated by using the formula described in DIN 53479 which is [2]:

$$Q_{PK} = \frac{m_1 Q_v}{m_2 - m_1}$$

Whereas,

Q_{PK} = Density of test sample, Q_v = Density of test liquid, m_1= weight of dry specimen, m_2= weight of wet specimen.

Calculated density values of all batches of visco-elastic foam are shown in table 2.

Table 2. Density values of visco-elastic foam samples.

Batch Number	m_1 (gram)	m_2 (gram)	Q_v (g/cm^3)	Q_{PK} (g/cm^3)
1. Visco-elastic foam with full formulation	0.110	0.856	1	0.147
2 .Visco-elastic foam with catalyst	0.332	2.023	1	0.19
3. Visco-elastic foam with blowing agent	0.206	1.291	1	0.189
4. Visco-elastic foam with additive	0.278	0.907	1	0.44
5. Visco-elastic foam with filler	0.268	1.229	1	0.278

3.2 ESTIMATION OF CREAM TIME, GEL TIME AND RISE TIME

Cream time is the time interval at which mixture turns creamy and starts to expand. It is the bench time of mixing and usually measured in seconds. It is measured by using a stopwatch and it shows time periods in which mixture of all reactants turns creamy. Gel time is the time interval at which mixture turns into the foam of a gel and it is due to the cross linking reactions occurring in the mixture. To test for gel touch the foaming mixture with spatula, a thread will form between spatula and foam. It is also measured in seconds. Gel time is normally more than the cream time. Rise Time is the time interval at which mixture starts to rise up. It can be in minutes or seconds depending upon the type of foam synthesized. It is very important in foam synthesis because it shows time at which reaction gets completed and process of formation of foam starts. Rise time is greater than both cream time and gel time [1].

3.3 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR) ANALYSIS

FTIR analysis of all the samples of foam was done by using Shimadzu IR Prestige-21/DRS-8000 with a resolution of 4 cm^{-1} with number of scans 50 and the instrument was run at transmittance mode. The data interval provided by the instrument for a resolution of 4 cm^{-1} is 1 cm^{-1}. The software used was IR Solution. All spectra were recorded from 4000 to 800 cm^{-1}. Spectra of all foam samples were obtained which indicates the various functional groups present in those samples.

4 RESULTS AND DISCUSSIONS

4.1 DENSITY MEASUREMENTS

There is a little variation in density values of all the samples of viscoelastic foam and this is due to the reason that all the ingredients are almost same in every batch of viscoelastic foam with very little variation in quantities of different reactants. In case of samples of VE foam with additive and filler the density value is 0.44 g/ cm^3 and 0.278 g/ cm^3 respectively. The density values of these two samples slightly differ from the density of other samples of visco-elastic foam; because when additive is used, density value becomes higher. Density values of all VE foam samples are almost same.

4.2 CREAM, GEL AND RISE TIME FOR VISCO-ELASTIC FOAM SAMPLES

Cream, gel and rise time of all samples of visco-elastic foam is measured. In first batch soft visco-elastic foam is formed in which cream time, gel time and rise time are 4 s, 6 s and 10 s respectively. Water is used as a blowing agent in this batch that's why mixture rises rapidly in a very less time. In second batch Cream time, gel time and rise time are 5 s, 6 s and 11 s respectively. In this batch there is a slight difference in the rise time as compared to the first batch and this is due to the reason that no blowing agent is used here and mixture starts to rise after the complete reaction of reactants.

In third batch Cream time, gel time and rise time are 7 s, 9 s and 17 s respectively. Now in this case there is a major difference in gel time and rise time as compared to other batches. This difference is due to the addition of filler as filler is added and also no blowing agent is used so mixing is required for more time and the ingredients will react with each other as the mixing continues. There is no rapid rise because the reaction will take time to complete due to the addition of filler and even it is observed that when the mixture take the form of gel then rise will not start at once but rise started after some seconds. In fourth batch Cream time, gel time and rise time are 3 s, 5 s and 10 s respectively. In this batch rise is rapid due to the use of water as physical blowing agent. In fifth batch Cream time, gel time and rise time are 2 s, 3 s and 7 s respectively. In this batch a rapid and fast rise is observed and this is due to the catalyst action, as catalyst enhances the rate of reaction without being the part of reaction so due to the addition of catalyst the rise time is very small and reaction gets completed in a very short time and after taking the form of gel mixture at once rise resulting in a fine and soft visco-elastic foam.

Measured values of cream time, gel time and rise time for all the samples of visco-elastic foam are represented graphically in figure 1, 2 and 3 respectively.

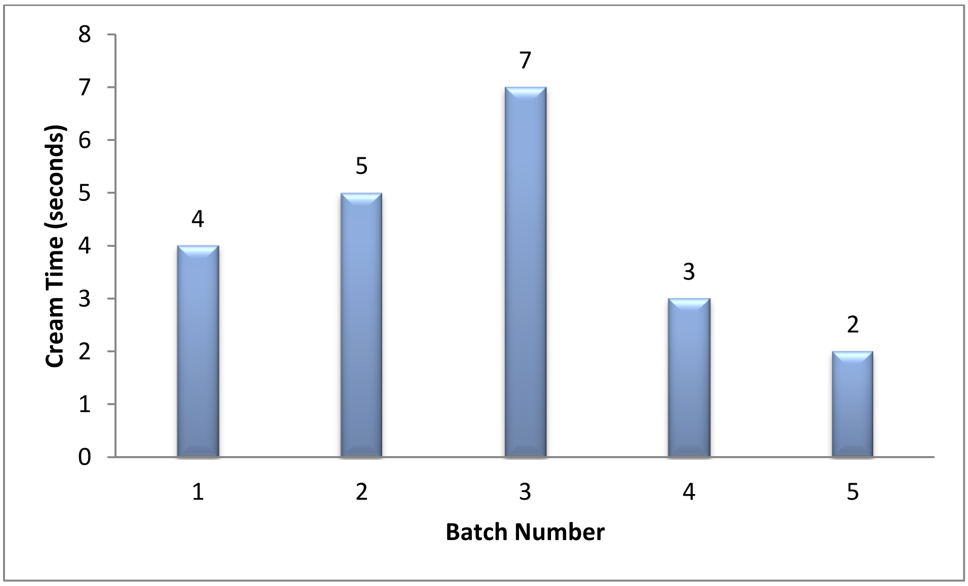

Fig. 1. Variation in cream time with batch number for visco-elastic foam samples.

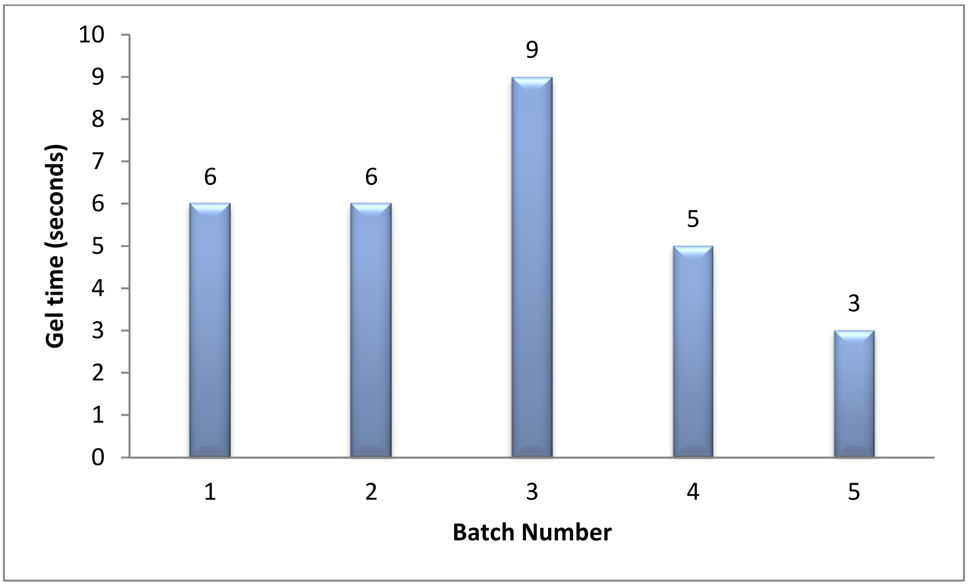

Fig. 2. Variation in gel time with batch number for visco-elastic foam

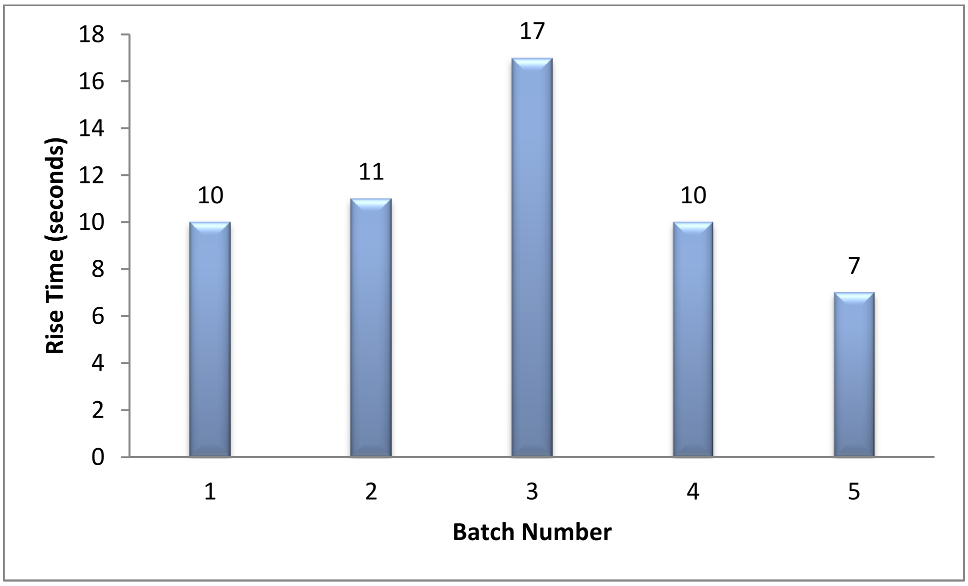

Fig. 3. Variation in rise time with batch number for visco-elastic foam

4.3 FTIR RESULTS

In spectra of the first sample the first peak comes at 3782.41 cm^{-1} which shows water contaminant and it is also present in the spectra of industrial sample. The peak at 3329.14 cm^{-1} shows the C-H stretch of alkynes and peak at 2879.72 cm^{-1} show C-H stretch of aliphatic alkanes. At 1710.86 cm^{-1} peak indicates saturated carboxylic acid derivatives with bond C=O. Peak at 1597.06 cm^{-1} shows aromatic rings and peak at 1525.69 cm^{-1} indicates the presence of nitro compounds N-O. Peak at 1361.74 cm^{-1} shows the presence of C-N amines and peaks at 1301 and 1288.66 cm^{-1} indicates the presence of aromatic ethers C-O which is due to polyether polyol. At 1095.57 cm^{-1} there is C-C stretch of alkanes and at 923.90 cm^{-1} peak shows C-H bend of alkenes. At 827.46 cm^{-1} and 758.02 cm^{-1} peak shows C-H bend of aromatic rings. Figure 4 shows the FTIR spectra of first sample of viscoelastic foam.

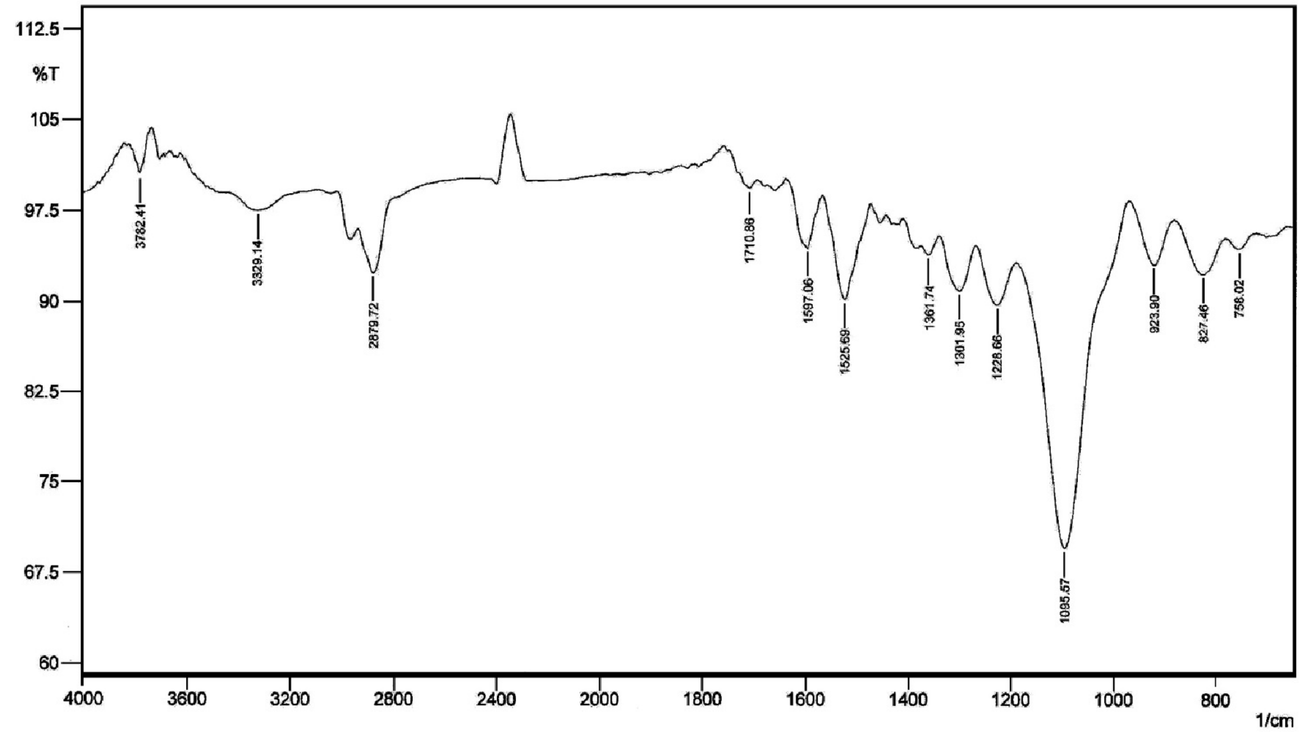

Fig. 4. FTIR spectra of viscoelastic foam (1^{st} sample)

FTIR spectra of viscoelastic foam sample in which catalyst is used is same as that of first sample and no major difference is observed. The only difference is that it is not showing any peak indicating water contaminant which shows that water is not used in synthesis of this sample. Peak at 3329.14 cm^{-1} shows C-H stretch of alkynes and peak at 2872.01 indicates C-H with specific bond methyl which shows the presence of MDI. Rest of the peaks in the spectra are same as the spectra of previous sample and show same functional groups and same frequency values indicating that the same chemicals are used in synthesis as of previous sample and use of catalyst do no effect the results of FTIR. Figure 5 shows the FTIR spectra of this sample.

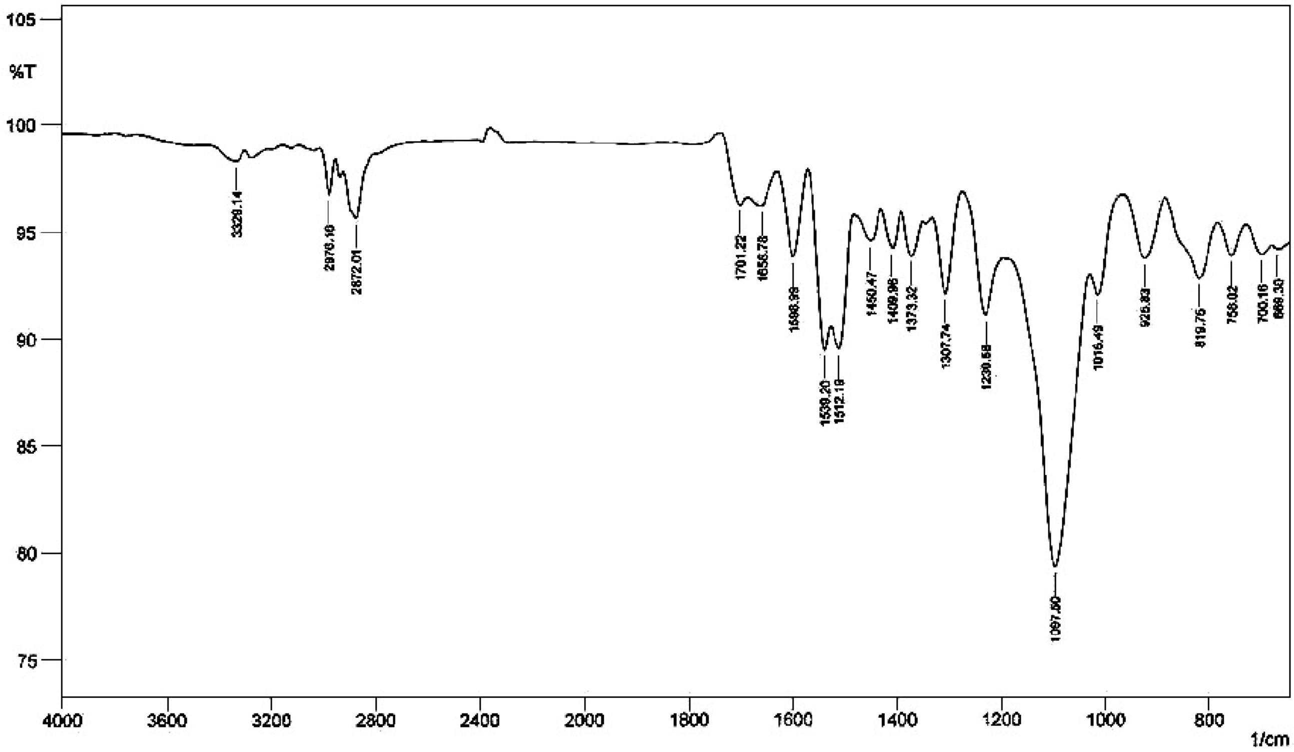

Fig. 5. FTIR spectra of viscoelastic foam sample in which catalyst is used

FTIR spectra of viscoelastic foam synthesized using a blowing agent is also same as spectra of previous samples showing that same chemicals are used. First three peaks shows the same C-H stretch and C-H with specific bonds. At 2870.08 cm^{-1} the peak shows C-H alkyl bond with methyl as a specific bond which is due to the presence of MDI. Peak at 1703.14 cm^{-1} show carboxylic acid derivatives and other rest peaks show aromatic rings, aliphatic nitro compounds, and ethers and in the end of spectrum all peaks show C-H bend of alkenes. Figure 6 shows the FTIR spectra of this sample.

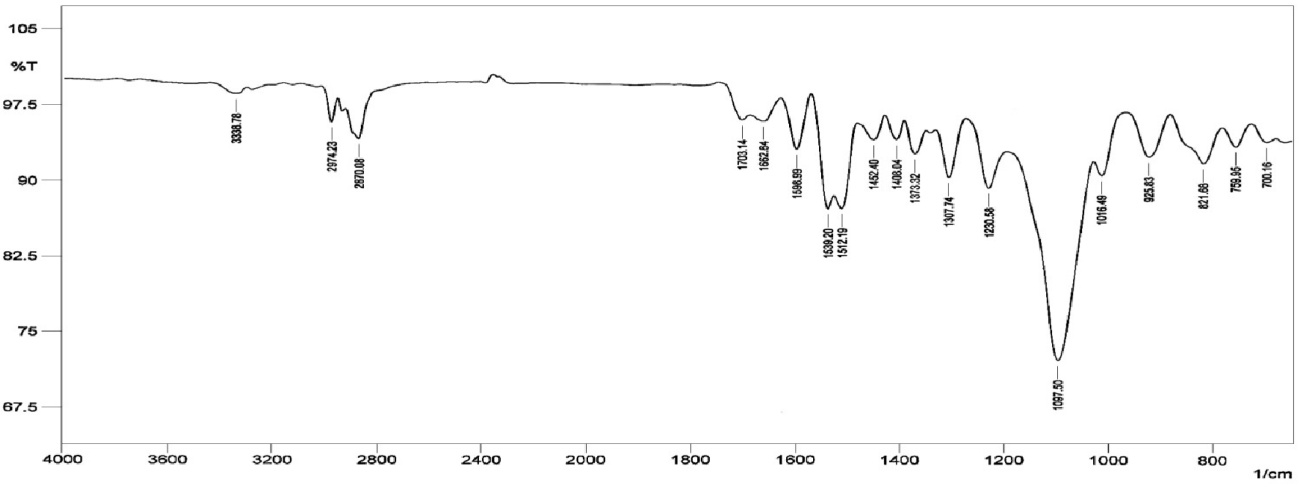

Fig. 6. FTIR spectra of viscoelastic foam sample with blowing agent

FTIR spectra of viscoelastic foam with additive is same as previous sample and no change is observed showing that addition of additive have no effect on FTIR results. All peaks in the spectra of this sample show same functional groups as in the previous samples. Additive used here is monol (benzyl alcohol) and at 1307.74 cm^{-1} peak shows the presence of C-O alcohols that justify the presence of benzyl alcohol in the sample that is used as an additive, rest of peaks are same. An FTIR spectrum of this sample is shown below in figure 7.

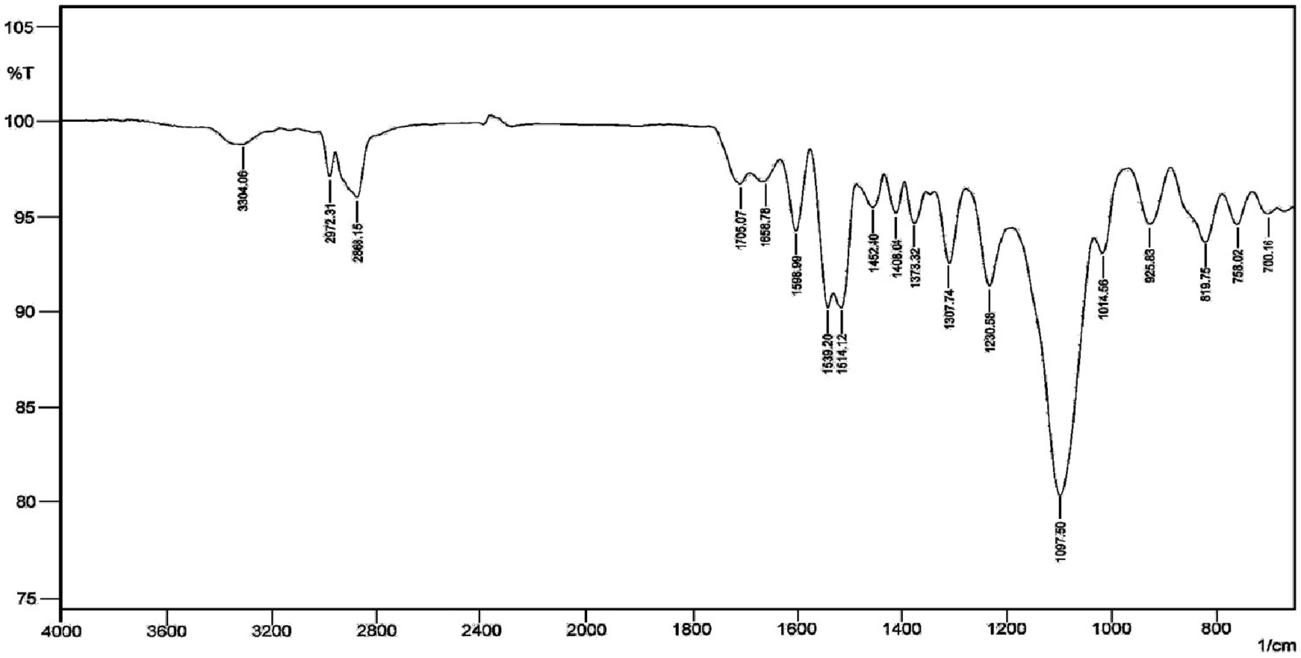

Fig. 7. FTIR spectra of viscoelastic foam sample with additive

FTIR of viscoelastic foam with filler exhibit the same results as of previous sample. As $CaCO_3$ is used as filler in this sample it has no effect on the FTIR spectra. There is a little difference in the values at which peak occurs but the functional groups of all the peaks are same as in the spectra of previous samples. In start the peaks show C-H stretch and bend with some specific bonds like methyl bond shown at 2868.15 cm^{-1}. Then there are different functional groups like carboxylic acid derivatives, aromatic and aliphatic nitro compounds, aromatic rings etc. FTIR spectra of this sample are shown below.

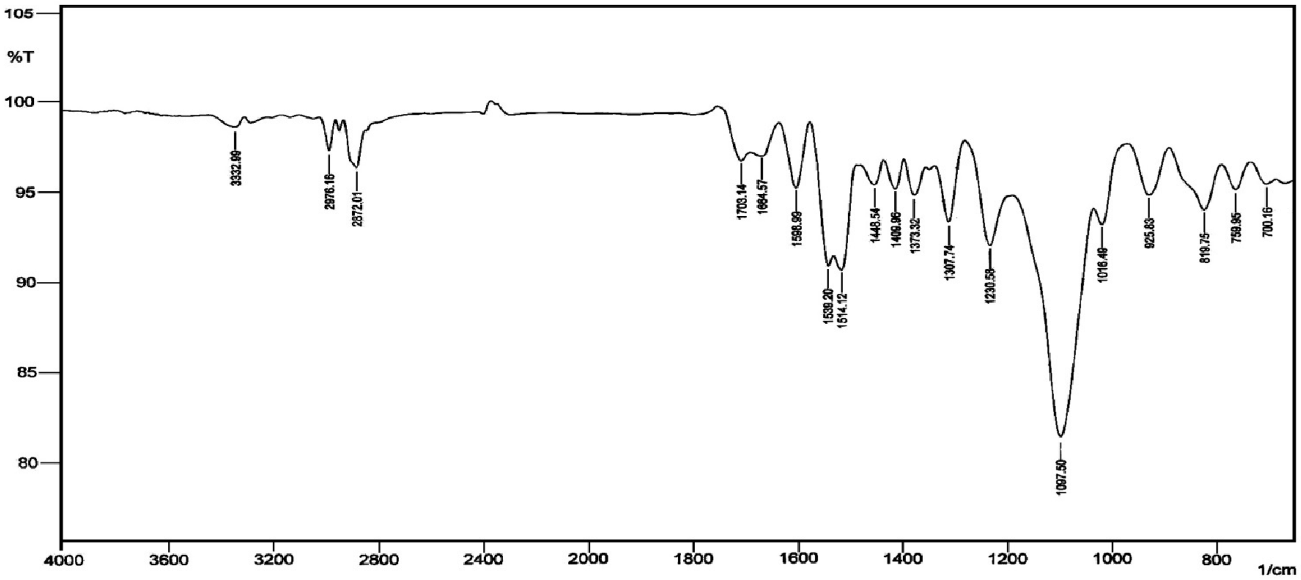

Fig. 8. FTIR Spectra of viscoelastic foam with filler

5 CONCLUSIONS

It can be concluded that:

- Visco-Elastic foam can be synthesized on lab scale using standard hand mixing procedures, as cup foaming was used in this research.
- Little variation in chemicals and their quantities can change the structure of final product.
- Addition of catalyst in foaming processes enhances the reaction speed resulting in rapid rise time.
- Use of blowing agent results in smaller value of rise time rise time because blowing agent causes the mixture to blow rapidly and reaction gets complete in very less time.
- Value of density can define the type of foam synthesized. On the basis of variation in density values of all the samples we can say that we have synthesized different grades of visco-elastic foam.
- FTIR results lead to conclusion that major reactants of foaming processes were present in all types of samples showing their respective peaks in spectra of every foam sample.

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