

# Enhancement of mechanical properties for reinforced Iraqi bentonite clay polyvinyl chloride composite using ultrasonic technique

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## Abstract

This research is depended an accurate style in preparing homogenous solution of PVC- Iraqi Bentonite clay composite adoption of careful balance mechanism in the selection of the ratio of solvent and mixing time according to the speed of mixing 450 rpm . Particle sizes selected for Bentonite clay is  $<75$  &  $<150\mu\text{m}$  and the calcination process was performed at 300, 700, and 900oC for 2hr. The Bentonite particles were mixed with known concentration of PVA solution at 80oC for half an hour, then dried, milled, and sieved for particle sizes as mention above. Mixture of PVC solution with reinforced Bentonite clay is used to prepare the composite under study. Effect of mentioned parameters on mechanical properties of PVC were performed by ultrasonic technique using frequency of 30 KHz. Addition of reinforced Bentonite generally enhanced the mechanical properties of PVC. Evidently the composite prepared from reinforced powder  $<75\mu\text{m}$  is a good medium for transfer ultrasound waves particularly at calcination temperature 300oC, that is mean it has a good stability against mechanical waves, so they can be used as a coated material for objects that we don't want them to be detected by sonar, while in the case of particles  $<150\mu\text{m}$  is not. Failure to obtain the same result at temperatures higher than 300oC and the granular size  $<150\mu\text{m}$  due to the presence strong intermolecular interaction that is responsible for reducing the elasticity of composite , in other words, leave it semi-brittle, i.e. not have good stability. Highest enhancement in the absorption coefficient appears at 900oC with preference to the specimen of particles  $<150\mu\text{m}$ ,and the composite becomes good absorber for ultrasound. Moreover the addition causing a reduction in compressibility, thus there is a small strain in the composite as a result of ultrasonic stress.

**Keywords:** PVC composite, Bentonite clay, Ultrasonic technique

## 1. Introduction

A composite is a structural material that consist of two or more constituents that are combined at a macroscopic level and are not soluble in each other. One constituent is called the reinforcing phase , which is mixing with materials that has coupling agents properties to prevent the collapse of the mechanical properties of a composite material ,and the other in which it is embedded is called the matrix. Inefficient industrial fillers (clays, black carbon and silica) used to reduce the cost of composite materials [1]. PVC in the form of powders, slurries, liquids and tablets. It has a wide range properties such as a wide range of colors, solid , rigid and stiff materials (with high viscosity) at room temperature , light weight with a good resistance to bases & acids , alcohol , oils , compound hydrocarbon aliphatic . PVC's relatively low cost, biological and chemical resistance and workability and can form easily have resulted in it being used for a wide variety of applications, such as insulation on electrical cables ,solid pipe manufacturing ,window frames and doors , bottles , containers , furniture industry [2] .

Bentonite is the rock composed essentially of clay minerals of the smectic, usually montmorillonite. Other smectic group minerals include hectorite, saponite, beidelite and nontronite. The theoretical formula for the structure mentioned above are  $(\text{OH})_4\text{Si}_8\text{Al}_4\text{O}_{20}\text{n}$  (interlayer)  $\text{H}_2\text{O}$ ,and the chemical formula for the montmorillonite group is  $(\text{Al},\text{Mg})_2(\text{OH})_2(\text{Al},\text{Si})_4\text{O}_{10}$ [3] . Bentonite has a high content of montmorillonite and a less amount of other clay minerals. Nowadays many different industrial branches such as ceramics, paper, rubber, wine, soaps and cleaning and polishing of compounds, and drug industries are using these materials, waste water purification and even into different

aspects of nano-technology because of nano sizes of its particles [4]. Bentonite group minerals show a colloidal structure in water, due to their internal structure and small particle size. They have a large adsorption capacity for polymer compounds due to their unique crystal structure [5].

The polymer concentration, molecular weight, hydrolysis degree of polymer, clay particle's size, shape, and surface charge, clay concentration in dispersion, clay's PH, and temperature are effective factors when clay particles interact with the polymers [6]. The aim of present work is to enhancement of mechanical properties including ultrasound velocity, density, absorption coefficient, shear viscosity, relaxation amplitude, relaxation time, compressibility, acoustic impedance, and bulk modulus of PVC by adding reinforced Iraqi bentonite clay.

## 2. Experimental and theoretical calculations

The work have been into six specimens depending on the certain selected of particles size (<75 & <150)  $\mu\text{m}$  and calcinations process (300,700, and 900)  $^{\circ}\text{C}$  for Iraqi Bentonite clay.

### 2.1 Preparation and testing reinforced Bentonite clay

The Bentonite particles were dispersed in distilled water to dispose from the salts and some of impurities that dissolved in water, and shaken extensively for 24hr at room temperature, after filtration, the specimen was dried at  $100^{\circ}\text{C}$  for 24hr by using the dryer type F. G. BODE&CO- Laboratory - Equipment - Hamburg-90. The chemical composition of Iraqi bentonite, which were supplied by state company of geological survey and minority in Iraq are listed in Table (1). The mineralogical analysis of the final powder of Iraqi Bentonite is listed in Table (2). The addition of PVA solution (PH~3) association as a coated layer covered the bentonite before applied it as filler. The mixture was mixed with heat treatment continued to get slurry form and to insure homogeneity with high viscosity (the mixing process was adopted according to the method of green land) [7], then dried, milled and sieved to particle sizes < 75, and <150 $\mu\text{m}$ .

### 2. Preparation PVC - reinforced Bentonite composite

A solution of 25g of PVC were prepared by using Sikloheksanon solvent in temperature of  $80^{\circ}\text{C}$  using a heater plate and mechanical mixer with feather glass type (Heidolph RZR 2050 electronic) and speed of mixing 450 rpm. By continuously mix we obtained a homogenous emulsion milky color. Incremental amount of reinforced Bentonite was added to prepared PVC solution after an investigation the case of emulsion and homogenized through a process of heating and mixing half an hour, as shown in Table (3), then we added the calcium stearate as stabilizer heat for certain quantity as defined in Table (3), and continued to mix that we have a brown emulsion. Then the material is poured into a glass template parallelogram. The specimen is kept in an incubator at  $30^{\circ}\text{C}$  for three days, and then moved to a container drying (we follow the same way to prepare the other specimens). After that the piece was prepared in the form of models of disks with a diameter 40mm for the purpose of examination. Mechanical properties were measured using ultrasonic technique at frequency of 30 KHz.

### 2.3. Theoretical calculations

The absorption coefficient ( $\alpha$ ) was calculated from Beer–Lambert law [8]:

$$A/A_0 = e^{(-\alpha x)} \dots\dots (1)$$

Where ( $A_0$ ) is the initially amplitude of the ultrasonic waves, (A) is the wave amplitude after absorption, and (x) is the thickness of the specimen. The relaxation amplitude of ultrasonic wave was calculated from the equation [9]:

$$D = \alpha / f^2 \dots\dots\dots (2)$$

Where (f) is the frequency. The method of measuring the speed of ultrasound wave was by calculating the thickness of the sample (x) and the time it takes inside the specimen (t) [10]:

$$v = x / t \dots\dots (3)$$

The acoustic impedance of a medium (Z) is a material property was calculated by equation [11]:

$$Z = \rho v \dots\dots\dots (4)$$

Where ( $\rho$ ) is the density. The bulk modulus (B) of a substance measures and the substance's resistance to uniform compression, it is defined as the pressure increase needed to decrease the volume; its base unit is the Pascal (Pa.) was calculated by equation [12]:

$$B = \rho v^2 \dots \dots (5)$$

Compressibility ( $\beta$ ) is a measure of the relative volume change of a fluid or solid as a response to a pressure (or mean stress) change, it was calculated by equation [13]:

$$\beta = (\rho v^2)^{-1} \dots \dots (6)$$

On the basis that all solids flow to a small extent in response to small shear stress, some researchers have contended that substances known as amorphous solids, such as glass and many polymers may be considered to have viscosity. This has led some to the view that solids are simply "liquids" with a very high viscosity; the viscosity of the samples was measured by using the equation [14, 15]:

$$\eta_s = 3 \alpha \rho v^3 / 8 \pi^2 f^2 \dots \dots (7)$$

The relaxation time ( $\tau$ ) was calculated from the equation [16] :

$$\tau = 4 \eta_s / 3 \rho v^2 \dots \dots (8)$$

### 3. Results and discussion

Ultrasonic velocity for all prepared specimens is higher compared with PVC as shown in Fig. 1, specified by specimen from reinforced powder <75 $\mu$ m at 300 $^\circ$ C. This attributed to the interaction causing association between the three types of molecules; polymer, solvent and bentonite molecules, so their will be entanglement action of high molecular weight of PVC to reinforced bentonite powder that can fills the spaces between polymer chains, and these entanglements enhanced the prepared composite to be a good medium for transferring ultrasonic waves. The velocity of ultrasound in the specimens prepared from reinforced powder <150 $\mu$ m is nearly constant and remain less than it is in the case of <75 $\mu$ m at all calcinations temperatures since the new lattice has not been good stability against ultrasonic waves as a result of not completely that specimens of 150 $\mu$ m can fills the whole spaces leaving somewhere that attenuated ultrasonic waves for improve relaxation times [17] as we will see later in Fig. 6. This behavior similar to that obtained by Boro Djardjevic for other materials [18]. The above result supported by the behavior of density as shown in Fig. 2 , where we find the specimens prepared from reinforced powder <75  $\mu$ m have the highest density than that of 150 $\mu$ m particularly at 300 $^\circ$ C.

The absorption coefficient for all prepared specimens is higher compared with PVC as shown in Fig. 3. This attributed that the attenuation of ultrasonic energy depends on shear viscosity, thermal conductivity, scattering, and intermolecular interaction. Thermal conductivity and scattering effects are known to be negligible [19], so the shear viscosity is the main cause of increasing absorption coefficient and as we look to the Fig. 4, especially obvious at 300 $^\circ$ C. High values at all calcinations temperatures attributed to the mechanism of hydrogen bonding attached to oxygen sites, which lead to increase the entanglement between filler molecules of size <75 $\mu$ m and polymer chains that randomly coiled so increasing shear viscosity [20], while in the case of 150 $\mu$ m the shear viscosity are nearly constant with temperature , because of an tied large grains within the lattice that leaving the specimen semi-brittle or not have good stability particularly in calcinations temperature higher than 300 $^\circ$ C [13,20]. This result supported by the behavior of variable bulk modulus verses calcinations temperature as we come to that later.

The high relaxation amplitude for both specimens prepared from reinforced powder <75 $\mu$ m and <150  $\mu$ m as shown in Fig. 5 associated with high absorption coefficient as shown previously, so that the absorbed molecules take time to relax to their equilibrium positions.

Effect of the calcinations process over the values of relaxation time after additives as shown in Fig. 6, because of the ultrasonic waves are propagated in the form of compression and rarefaction when interact with polymer molecules, so as a pure specimens its molecules take a time to relax to their original position against sound waves ,while the additives restrict these molecules that increase the relax time particularly in specimens prepared from reinforced powder <75 $\mu$ m at 300 $^\circ$ C because it has stability higher than that of <150 $\mu$ m .

The general decreasing in compressibility after additives reinforced Bentonite of different sizes compared with PVC and improve it with calcinations temperature as shown in Fig. 7, is attributed to the inversely relation between velocity and compressibility, since the polymer molecules be closer to each other and there will be interactions of strongly bonds obtained as a result of increasing calcinations temperature. Effect of addition reinforced Bentonite of different sizes on the values of specific acoustic impedance as shown in Fig. 8 interpreted on the basis that the increase in velocity caused an increase in specific acoustic impedance largest in specimens prepared from reinforced powder  $<75\mu\text{m}$  particularly at  $300^\circ\text{C}$ , because of high stability against sound waves [21,22]. Bulk modulus also increasing after additives as shown in Fig. 9. This attributed to the fact that compressibility is inversely related to the Bulk modulus by means of Eq.5 and Eq.6, when compressibility decrease bulk modulus must be increasing particularly for specimens prepared from reinforced powder  $<75\mu\text{m}$  at  $300^\circ\text{C}$ .

#### 4. Conclusions

From the experimental results we can show that the mechanical properties of composites can be changed using the reinforced Bentonite.

- 1- The addition of reinforced bentonite generally enhanced the mechanical properties of PVC.
- 2-The composite prepared from reinforced powder  $<75\mu\text{m}$  is a good medium for transfer ultrasound waves particularly at calcinations temperature of  $300^\circ\text{C}$ , that is mean it has a good stability against mechanical waves, so they can be used as a coated material for objects that we don't want them to be detected by sonar, while in the case of particles  $<150\mu\text{m}$  is not.
- 3- Failure to obtain the same result at temperatures higher than  $300^\circ\text{C}$  and the granular size  $<150\mu\text{m}$  due to the presence strong intermolecular interaction that is responsible for reducing the elasticity of composite, in other words, leave it semi-brittle, i.e. not have good stability.
- 4- Highest enhancement in the absorption coefficient appears at  $900^\circ\text{C}$  with preference to the spacemen of particles  $<150\mu\text{m}$ , then the composite becomes good absorber for ultrasound.
- 5-The addition of reinforced Bentonite causing a reduction in compressibility and thus there is a small strain in the composite as a result of ultrasonic stress.

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Table 1. Chemical composition of used Bentonite.

Oxide	Wt%	Oxide	Wt%	Oxide	Wt%
SiO <sub>2</sub>	56.77	MgO	3.42	SO <sub>3</sub>	0.59
Al <sub>2</sub> O <sub>3</sub>	26.2	Na <sub>2</sub> O	1.11	Cl	0.57
Fe <sub>2</sub> O <sub>3</sub>	5.12	K <sub>2</sub> O	0.6	L.O.I	0.49
CaO	4.48	P <sub>2</sub> O <sub>5</sub>	0.65	( loss of ignition)	

Table 2. Mineralogical analysis of used Bentonite.

Minerals	Percentage %
Montmorillonite $[\text{OH}]_2\text{Al}_2\text{SiO}_{10}$	79
Attapulgate $\text{Mg}_5\text{Si}_8\text{O}_{20}[\text{OH}]_28\text{H}_2\text{O}$	7
Apatite $\text{Ca}_5[\text{Fe, Cl}] \text{P}_3\text{O}_{13}$	5
Calcite $\text{CaCO}_3$	5
Gypsum $\text{CaSO}_42\text{H}_2\text{O}$	2
Halite $\text{NaCl}$	1
Quartz $\text{SiO}_2$	1

Table 3. The ratios and type of components, calcinations temperature and particle size of filler powder.

Sample NO.	Filler preparation condition (reinforced)					PVC preparation condition (matrix)				
	Particle size ( $\mu\text{m}$ )	Calcinations temperature $^\circ\text{C}$	Calcinations time (hr)	Filler(Bentonite)wt%	PVA wt%	PVC wt%	Ratio Solvent (gm :ml)	Stabilizer %	Mixing temperature $^\circ\text{C}$	Mixing time (hr)
A1	<75	900	2	5	0.5	95	1: 2	1	85	2.5
A2	<150	900	2	5	0.5	95	1: 2.5	1	85	3.5
B1	<75	700	2	5	0.5	95	1: 3	1	85	4
B2	<150	700	2	5	0.5	95	1: 2.5	1	85	3.5
C1	<75	300	2	5	0.5	95	1: 2	1	85	3.5
C2	<150	300	2	5	0.5	95	1: 2.5	1	85	2.5

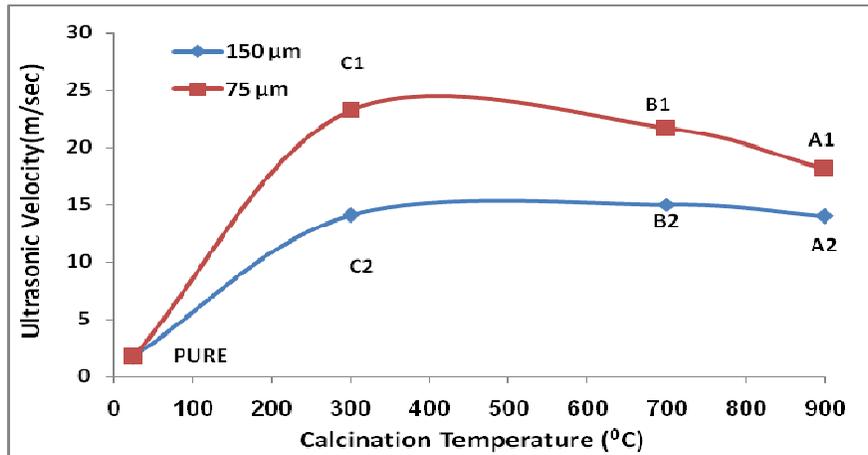


Figure 1. Plot of ultrasonic velocity versus calcination temperature.

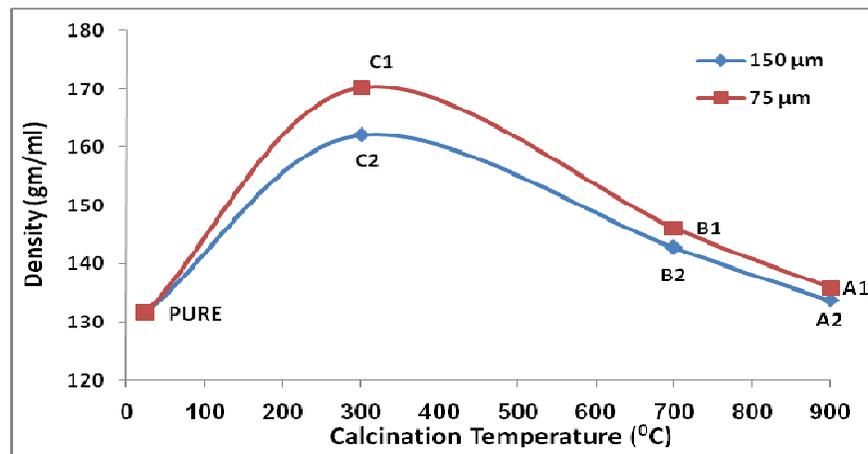


Figure 2. Plot of density versus calcinations temperature.

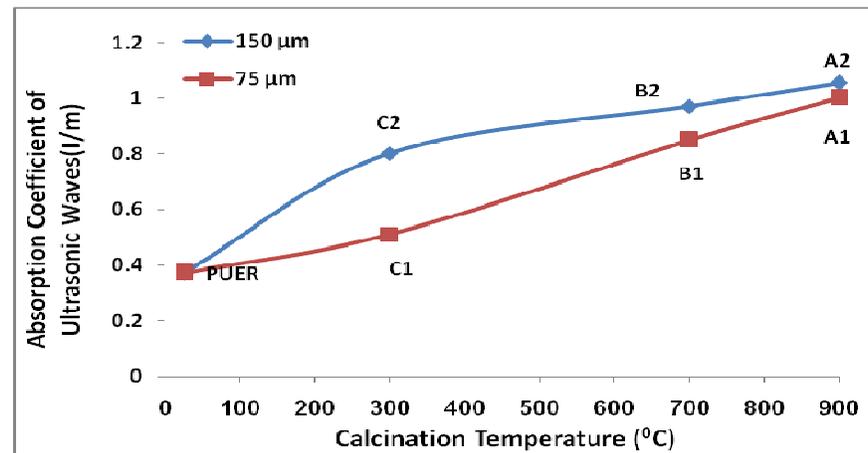


Figure 3. Plot of absorption coefficient of ultrasonic waves versus calcinations temperature.

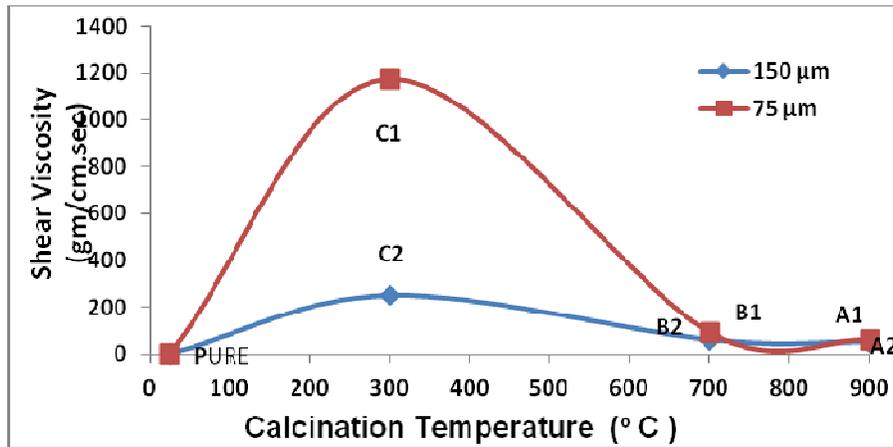


Figure 4. Plot of Shear viscosity versus of calcination temperature.

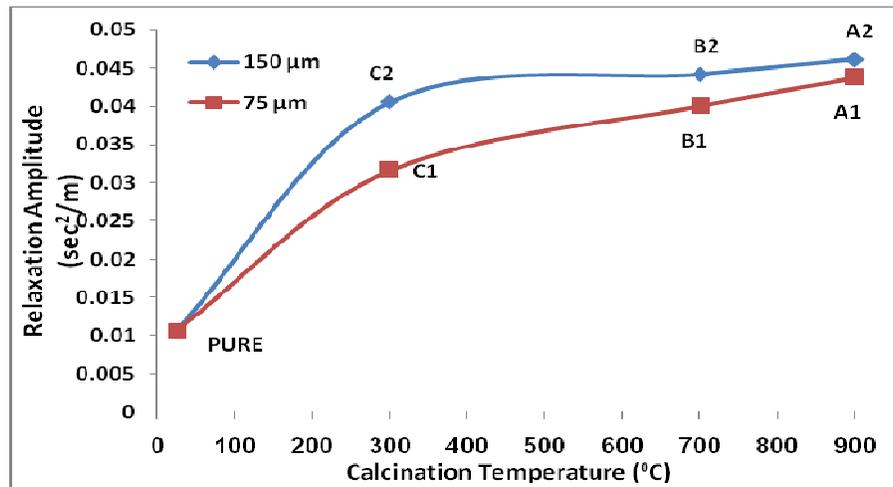


Figure 5. Plot of relaxation amplitude versus calcination temperature.

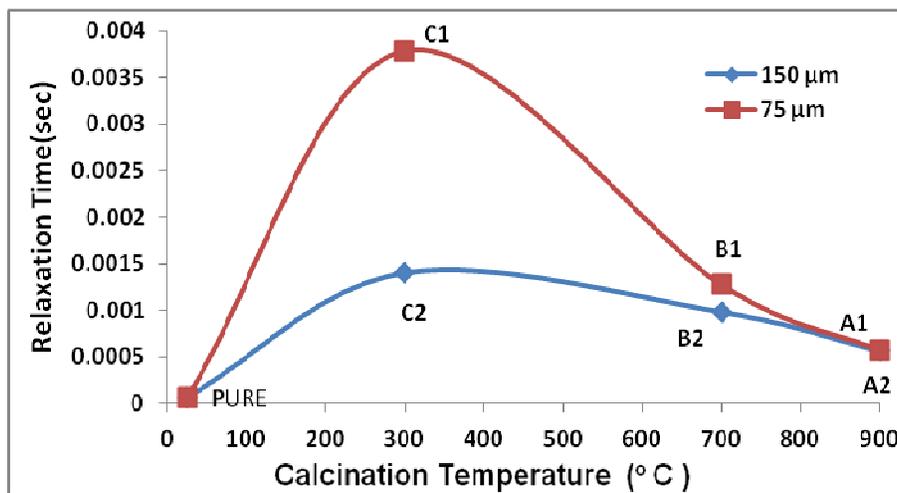


Figure 6. Plot of relaxation time versus calcination temperature.

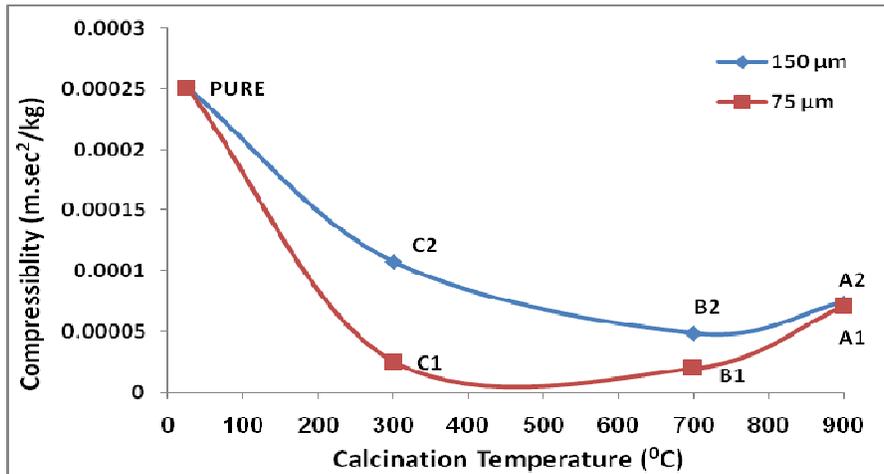


Figure 7. Plot of compressibility versus calcination temperature.

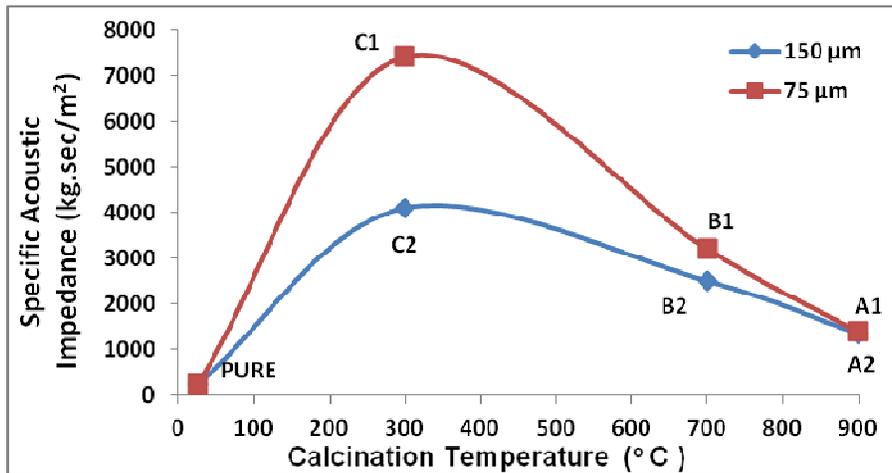


Figure 8. Plot of specific acoustic impedance versus calcinations Temperature.

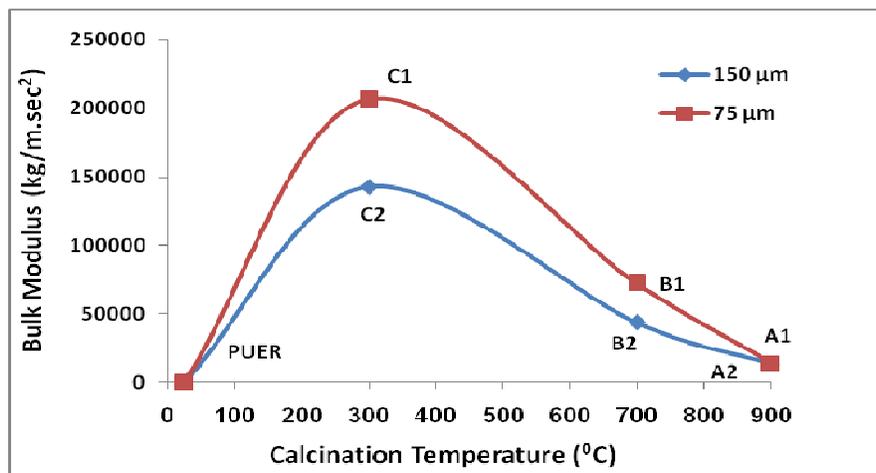


Figure 9. Plot of bulk modulus versus calcinations temperature.