

## **EFFECT OF PRESSURE ON ELASTIC PROPERTIES OF CHITOSAN**

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### **ABSTRACT**

The work focused on studying the elastic properties of chitosan composite system. The samples were prepared in form of compressed pellets. The structure and elemental analysis of chitosan was characterized using Fourier Transform Infrared Spectroscopy (FTIR). The FTIR spectra have been recorded in the spectral range from 280-4000cm<sup>-1</sup>. The velocity of sound of chitosan is obtained by the ultrasonic pulse-echo measurements, at room temperature. Both longitudinal and transverse velocities of chitosan are pressure dependence. From the value of density, longitudinal and shear ultrasonic velocity, the value of longitudinal modulus (L), shear modulus (G), bulk modulus (K), Young modulus (E) and Poisson ratio ( $\sigma$ ) were calculated. The sound velocity increases as the density and pressure increase. The elastic modulus and Poisson' ratio show increasing trend as the pressure of sample increases. The elastic modulus increases due to the compactness of the pressed sample. As the pressure and density increase, the atoms in chitosan became more compact thus the ultrasonic waves are dispersed between the atoms resulting in more propagation of wave.

*Keywords: chitosan; elastic modul; FTIR, pressure;*

### **INTRODUCTION**

Chitosan is a modified natural carbohydrate polymer derived from chitin, which is the structural element in the exoskeleton of crustaceans (crabs, shrimp, etc). The primary unit in the chitin polymer is 2-deoxy-2-(acetylamino) glucose. These units are combined by, 1-4 glycosidic linkages, forming a long chain linear polymer. Removal of most of the acetyl groups of chitin by treatment with strong alkalis yields chitosan.

Chitin and chitosan are considered ecologically conservation materials on the earth and it has many practical applications. Chitin and chitosan have been investigated for practical applications such as biomedical and agricultural purposes [1]. The aim of this work is to study the effect of pressure on the elastic moduli of pressed chitosan pellets using pulse-echo technique.

## **EXPERIMENTAL DETAILS**

### *Preparation of sample*

Chitosan obtained from Polymer Chemistry Lab, Chemistry Department, Universiti Putra Malaysia with ~ 88% degree of deacetylation. The powdered chitosan were heated at 303 K for 3 hours to remove moisture. Required amount of chitosan were weighed using digital balance having an accuracy  $\pm 0.0001$  g. The chitosan powder was pressed into pellets and to obtain even and parallel surfaces, the pellets were polished lightly to avoid crack in the sample with sand paper. Their thickness was about 0.8 cm.

### *Density Measurement*

Density of all pellets was calculated using geometrical technique applying the relation

$$\rho = \frac{m}{v} \quad (1)$$

where  $\rho$  is the density of pellet, m is the mass of pellet in gram and v is the volume of the pellet which was calculated using the equation for volume of cylinder.

### *Ultrasonic velocity measurement*

The ultrasonic measurement was carried out at room temperature using MBS8000 Ultrasonic Data Acquisition System with a transducer having resonating frequency of 5 MHz (both longitudinal and shear). The pulse section generates electrical pulses that are converted into ultrasonic signals using matched transducers. The ultrasonic pulse travels through the specimen bonded to the transducer and echo is registered each time and returns to the transducer. The ultrasonic wave velocity can be calculated as given in the following equation:

$$v = \frac{2x}{\Delta t} \quad (2)$$

where x is the sample thickness and  $\Delta t$  is the time interval. The measurements were repeated three times to check the reproducibility of the data.

### *FTIR studies*

Fourier Transform Infrared Spectrometer was used to measure the vibrational spectra of the investigated samples. The samples were measured using the KBr disc technique at room temperature in the wave number range 280-4000  $\text{cm}^{-1}$ .

## **RESULTS AND DISCUSSION**

The infrared spectra of chitosan were recorded in the wavenumber region 280-4000  $\text{cm}^{-1}$  as shown in Figure 1. The band in the range 3500-2500  $\text{cm}^{-1}$  is corresponding to the

vibrations of OH, NH and CH groups. The bands that appeared are at  $3295\text{ cm}^{-1}$  which is the characteristic of OH group and  $2877\text{ cm}^{-1}$  represents alkyl C-H stretch. The complex band in the range  $1690\text{-}1300\text{ cm}^{-1}$  corresponding to absorption of carbonyl and amide groups (Amide I band at  $1641\text{ cm}^{-1}$  and Amide II band at  $1585\text{ cm}^{-1}$ ). These two bands were assigned to N-H symmetric and asymmetric stretching vibration of amide group. Moreover, these spectra can be influenced by parameters such as the deacetylation percentage or crystallinity as chitosan is essentially produced from chitin by a deacetylation reaction. The IR spectra of chitosan correspond to a convolution of specific signals specific to both carbohydrates and absorption due to amine and amide functions. A shoulder is first observed at  $1641\text{ cm}^{-1}$ , which corresponds to the overlap of an Amide I band due to the residual acetylated residues and  $\text{-OH}$  bands corresponding to the pyranose structure. A transition corresponding to the Amide II band can be detected by the presence of a strong band at  $1585\text{ cm}^{-1}$ .  $\text{CH}_3$  deformation occurred at  $1375\text{ cm}^{-1}$  and this peak indicates incomplete deacetylation of chitin. The strong absorption band between  $800$  and  $1200\text{ cm}^{-1}$  is the characteristic of pyranose rings of chitosan. The band that appeared is at  $1028\text{ cm}^{-1}$  [2].

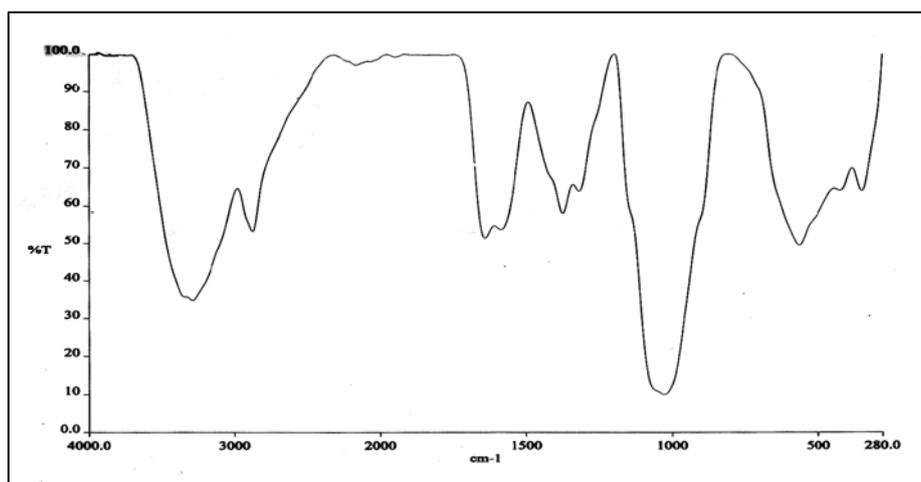


Figure 1: FTIR spectra of chitosan

The transit time and density ( $\rho$ ) of all pressed chitosan pellets studied are reported in Table 1. It can be seen that the density increases gradually with the pressed pressure. The density of the pellets ranged between  $1237$  to  $1244\text{ kgm}^{-3}$ . The dependence of density on pressure is in agreement with the mass of pellets. The thickness of the pellets is constant. In order to have a constant thickness, the mass of chitosan was increased. Thus, affects the density of the pellets.

Table 1: Measured transit time, density ( $\rho$ ), longitudinal ultrasonic velocity ( $V_l$ ) and shear ultrasonic velocity ( $V_s$ ) of chitosan

Pressure (tonne)	Transit time ( $\mu$ s)		$\rho$ (kg/m <sup>3</sup> )	$V_L$ (m/s)	$V_S$ (m/s)
	$V_L$	$V_S$			
2	9.9	17.5	1237	705	401
3	9.8	17.3	1238	718	404
4	9.6	17.2	1241	730	407
5	9.5	17.1	1242	737	410
6	9.3	16.8	1244	756	418

Figure 2 shows both longitudinal ( $V_l$ ) and transverse ( $V_s$ ) ultrasonic velocity of chitosan pellets. It is clear from the figure that both ultrasonic velocities increase with increasing of pressed pressure. The regular and continuous increase in ultrasonic velocities (Table 1) can be explained on the fact that when the pressure increased, the atoms in chitosan become more compact. Hence, it is easy for the ultrasonic wave to disperse inside the chitosan structure. The speed of waves depends on the hardness, compressibility and density of samples. However, all three of these factors depend also on the condition of the sample, such as the existence of cracks and stress. [3]

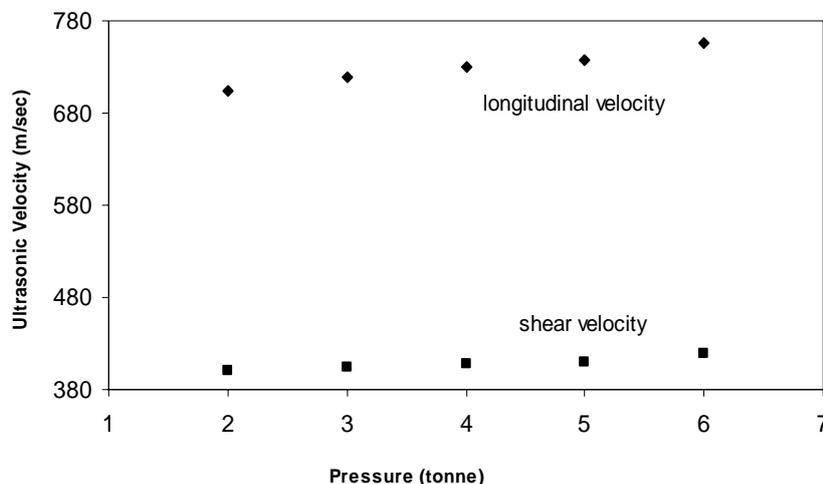


Figure 2: Variation of ultrasonic velocity with pressure of pressed chitosan pellets

Figure 3 shows the variation of the elastic moduli data of chitosan with different values of pressed pressure. Table 2 compiles the longitudinal modulus (L), shear modulus (G), Young's modulus (E), bulk modulus (K) and Poisson's ratio.

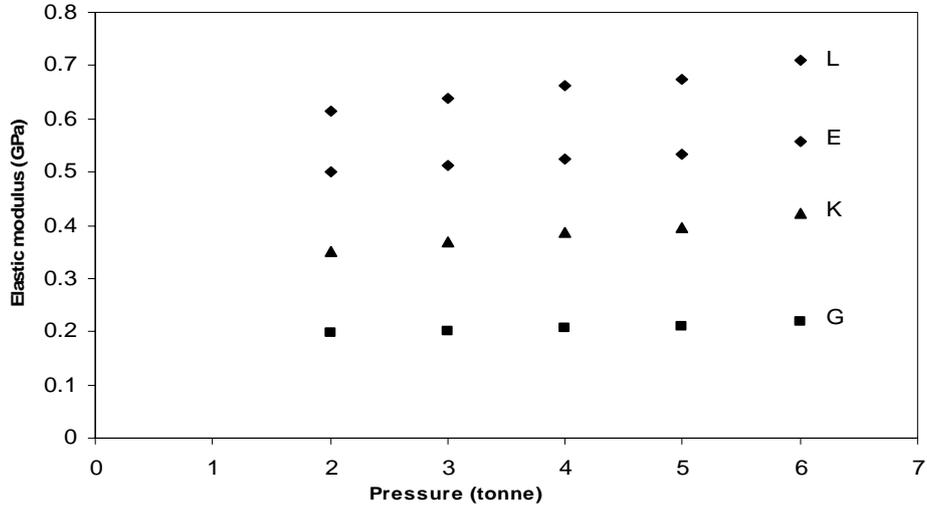


Figure 3: Elastic moduli longitudinal ( $L$ ), shear ( $G$ ), Young's ( $E$ ) and bulk modulus ( $K$ ) vs. pressure of pressed chitosan pellets

The room temperature of elastic moduli and Poisson's ration of chitosan pellets were calculated by means of expression [4]

$$L = \rho V_l^2 \quad (3)$$

$$G = \rho V_s^2 \quad (4)$$

$$K = L - \frac{4}{3}G \quad (5)$$

$$E = 2G(1 + \sigma) \quad (6)$$

$$\sigma = \frac{L - 2G}{2(L - G)} \quad (7)$$

where  $\rho$  is the density,  $V_l$  and  $V_s$  are the longitudinal and transverse ultrasonic respectively.

The results from Table 2 indicate that the elastic moduli increase with increasing of pressure from 2 to 6 tonne. Longitudinal modulus ranged from 0.615 to 0.711 GPa, shear modulus from 0.198 to 0.217 GPa, Young's modulus from 0.501 to 0.556 GPa, and bulk modulus from 0.350 to 0.421 GPa. The large difference between  $L$  and  $G$  arises from volume effects. The change in volume due to compressions and expansions involved in longitudinal strains is pronounced while no change in volume is involved in shear strains. However we observe that both plots have almost the same characteristic. [4]

Table 2: Elastic moduli longitudinal ( $L$ ), shear ( $G$ ), Young's ( $E$ ) and bulk modulus ( $K$ ) and Poisson's ratio of pressed chitosan pellets

Pressure (tonne)	L (GPa)	G (GPa)	E (GPa)	K (GPa)	$\sigma$
2	0.615	0.198	0.501	0.350	0.261
3	0.638	0.202	0.513	0.369	0.268
4	0.661	0.206	0.524	0.387	0.274
5	0.675	0.209	0.533	0.396	0.276
6	0.711	0.217	0.556	0.421	0.280

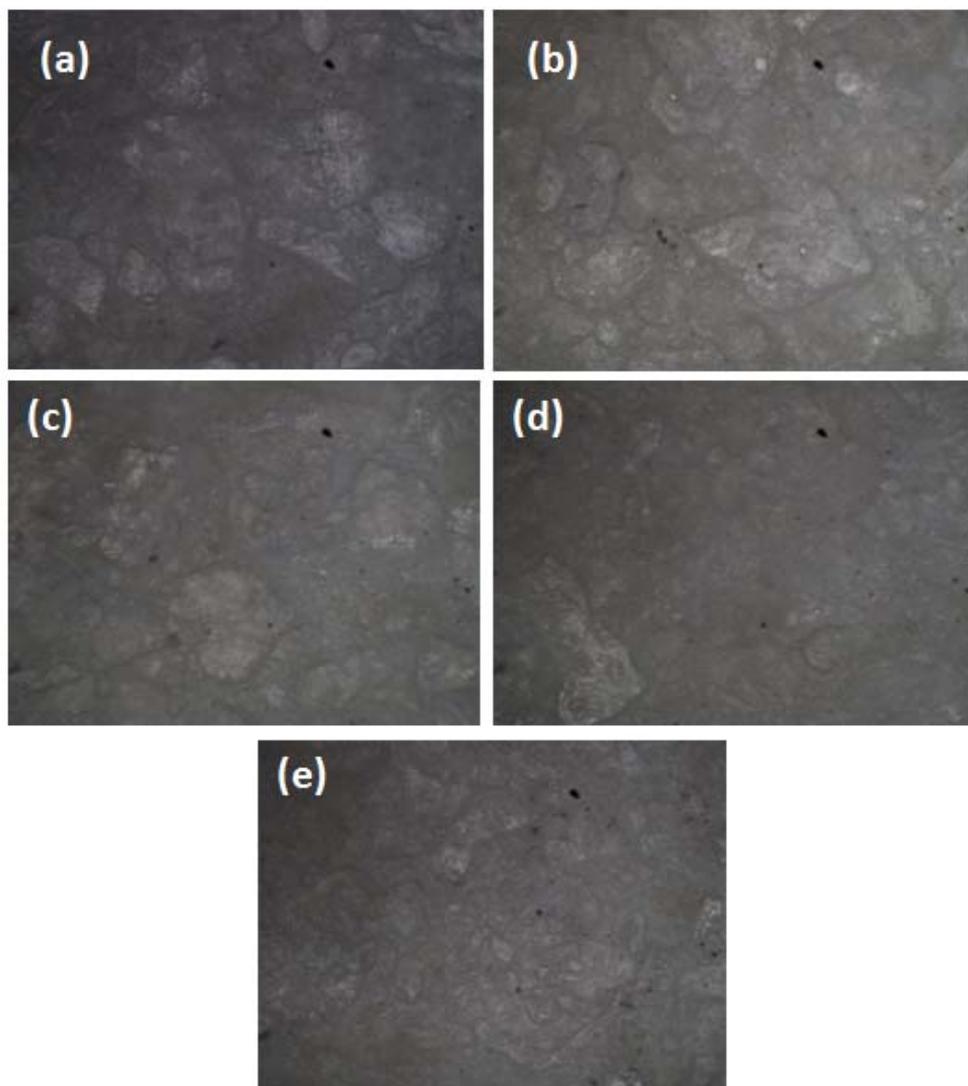


Figure 4: Optical microscope image for chitosan with various pressed pressure (a) 2tonne (b) 3 tonne (c) 4 tonne (d) 5 tonne (e) 6 tonne

From Figure 4 (a) to (e), the porosity of the pellets can be observed. The distribution of waves depends on the split or crack in sample. Cracks in the sample will increase the porosity between particles in chitosan pellets. Porosity will lower the value of Young's modulus. As the pressure and density increase, the atoms in chitosan became more compact. The holes between the atoms is reduced thus the ultrasonic waves are easily dispersed between the atoms resulting in more propagation of wave.

Poisson's ratio has been calculated using Equation (7). It can be seen on Figure 5 Poisson's ratio increased from 0.261 to 0.280 when the pressure of the pressed samples increases. When the density of chitosan increases, the speed of waves also increased. The relationship between the density of chitosan and waves across the sample is linear. When the density of the pellets is high, the Poisson's ratio will increase and therefore, the compressive strength of the sample also increases. [5]

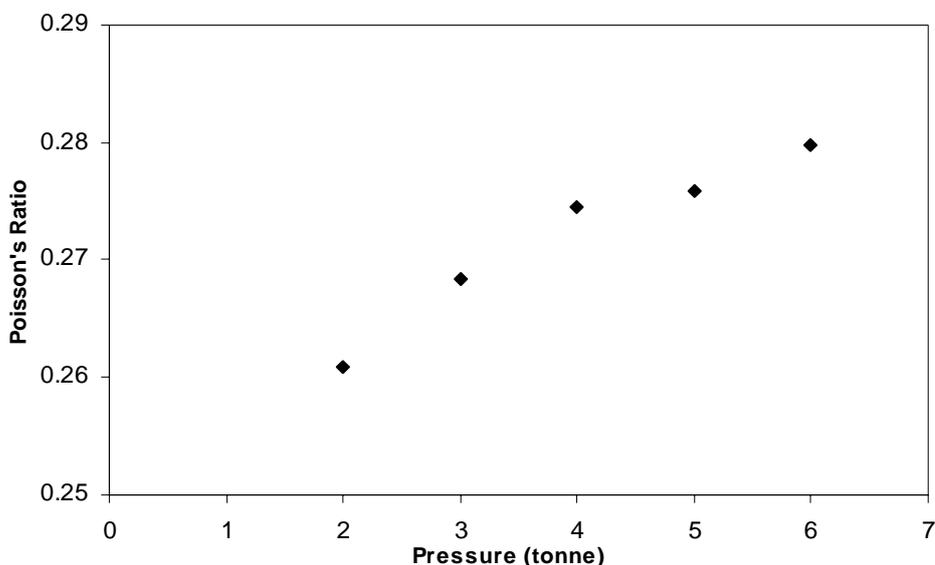


Figure 5: Variation of Poisson's ratio with pressure of pressed chitosan pellets

### CONCLUSION

The elastic moduli of chitosan increase with increasing pressure. Large difference between longitudinal and shear modulus is because of volume effects. The change in compressions and expansions involve in longitudinal strain while no volume change in shear strain. The Poisson's ratio of chitosan also increases as the pressure increase due to the increasing of density and therefore the compressive strength of the sample increase.

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