Synthesis, Characterization and USW Sensor of PEO/PMMA/PVP Doped with Zirconium Dioxide Nanoparticles

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Research Article

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Abstract

Piezoelectric phenomena is very important for various applications such as ultrasound waves (USW) sensor and pressure sensors. The raw materials from polyethylene oxide (PEO), Poly(methyl methacrylate)(PMMA) and poly(N-vinyl pyrrolidone) (PVP) polymer blends were doped with 0.02,0.04 and 0.06 wt% from zirconium dioxide (ZrO_2) nanoparticles (NPs) as a nanocomposites (NCs). The resultant films were prepared by dry casting method. The optical microscope (OM) and scanning electron microscopy (SEM) were used to satisfied from the SiO_2NPs diffusion in the blend. The SiO_2NPs were good diffused inside the blend with some weak aggregation happened in the high content of NPs. The composite films were diagnosed by FTIR. The USW properties were measured for k1 spaceman with various frequencies (25,30,…,45) MHz. The USW coefficients were clearly effected by the frequency varied. The resulted composite film was succeeded to be used as USW sensor, also these findings could help to realize these new NCs as promising materials for wide applications, such as backlight, car glass and UV filters.

1. Introduction

The piezoelectric effect or US sensor, known as the direct piezoelectric effect, is an electrical generating phenomena which occurs when a material is exposed to external pressure, mechanical stress or applied force. PEO is a one type of polymers that synthesis from polyether polymer (PE) [1]. PEO has a many important applications in treatment of water, papers industries, physical, medical and engineering [2]. PMMA is a tough and strong, lightweight polymer. It has a density of (1.17–1.20 g/cm^3), that is less than half that of glass [3]. PMMA has good impact strength, higher than both polystyrene and glass [3]. PVP is a one kind of vinyl polymers, PVP powder has white color, stable in different temperature ranges, hygroscopic, and good solubility in water. It has a good ability to complexes form with various materials [4]. Zirconium dioxide (ZrO_2) has distinguished physical and chemical properties such as heat and chemical resistance, high hardness and strength and catalytic activity characterization, furthermore its mostly used in catalysts industrializations, piezoelectricity, solid fuel batteries[5]. ZrO_2 properties change by very small amounts [5]. ZrO_2 have highly conditioned on the diameters of NPs [6]. The smallest ZrO_2NPs diameter usually absorbs the photon and have peaks near to UV region, but largest domain cause scattering increases and have broaden and shift to longer IR shifting wavelengths [7,8]. In the present work, the (PEO/PMMA/PVP)/ ZrO_2 NCs were prepared as a solutions and films by simple casting of solution method with various ratios of ZrO_2NPs. The structural and optical properties of the composite materials were studied. The structural properties include OM and SEM. The k1 spaceman was diagnosed by FTIR.

2. Experimental Study

2.1 Materials: PEO (6000 Mw and 99.8% assay purity) purchased from Reagent World, PMMA (5000 Mw and 99 % assay purity), purchased from DIDACTIC, PVP (Mw ˜ 40000 and 99.9% purity) purchased from Central Drug House, and ZrO_2NPs purchased from (US Research Nanomaterials, Inc.) with average grain size (40 nm) and assay purity (99.9%).

2.2 Synthesis of NCs: Polymers NCs films were prepared by mixing of the raw materials in (50 mL) deionized water (DI) in a glass beaker at (60 ^{o}C) hot plate magnetic stirrer. The process continued for (1 hrs.) to getting on homogenous mixture. The NPs were respectively added as (0.02, 0.04 and 0.6) wt.% as shown in Table (1), for (3 hrs.) then the mixture casted in (5 cm Petri dish) and leaved for two weeks to dry. By digital micrometer, the samples thickness were in the range between (80-95) µm.

Table 1. The method of purifications of (PEO/PMMA/PVP)/ZrO_2 NCs.

Sample ID	Ratio of Weight Percentages %				Mixing Time h
	PEO	PMMA	PVP	NPs	
k0	0.600	0.200	0.200	0.00	1
k1	0.588	0.196	0.196	0.02	3
k2	0.576	0.192	0.192	0.04	3
k3	0.564	0.188	0.188	0.06	3

3. The Om, Sem And Ftir Images:

Figures (1 and 2) represent the OM and SEM photomicrographs of k0,k1,k2 and k3 samples at (100X) magnification power. Figure (1-A) shows that the PEO/PMMA/PVP blend has acceptable and homogenous dissolving. The (B, C, and D) in the same figure show that the diffusion of ZrO_2NPs in the blend. The some weak agglomerations of NPs were clearly appeared in (C and D). The explanation of that related to the interaction that happen among ZrO_2NPs because the high value of surface to volume ratio. Scanning electron micrograph (SEM) is generally used to study the compatibility among various constituents of the polymer electrolytes. The SEM images of the polymer composites were depicted in figure (2). The micrograph indicates with the loaded of ZrO_2NPs, the surface clearly shows a uniformly distributed area. The micro structural observation is good agreement with OM results that have referred an improvement in the amorphous phase with the loaded of ZrO_2NPs. Furthermore, the SEM images refer to good diffusion of NPs, homogeneity and surface roughness of raw material/ZrO_2NPs composites.

Figure (3) represents the FTIR spectrum of k1 spaceman in the (500-4500) cm^{-1} wave number. From figure, we notice that, the chemical functional groups that respectevly appear in the computed optical range related to alcohol/phenol (OH) stretching appears in (3284.56 cm^{-1}), (C=C) stretching appears in (1662.29 cm^{-1}), the symmetric bending of CH_2 appaers in (1437.15 cm^{-1}), the CH wagging appears in the

(1289.84 cm^{-1}) and (C-O) stretching related to primery alcohol appaers in (1099.92 cm^{-1}). The (OH) wagging appears in (540 cm^{-1}) related to ZrO_2NPs.

4. The Usw Characterization

The morphological propertied were carried out using Nikon, Olympus model 73346 camera. The spaceman was characterized using FTIR (Vertex 701,Bruker) in the spectral range (4000 – 400) cm^{-1}. The USW measurements were practically made using (SV-DH-7A/SVX-7) at various frequencies. The USW were applied for testing in the region between the sender and receiver. The receiver converts US pulses to the electrical pulses then received by oscilloscope. The apparent signal in 1st. channel contains positive peak represent incident USW or initial amplitude (A_o) and the negative part in the 2nd. channel refers to receiver amplitude (A).

5. Results And Discussion

The density of PEO/PMMA/PVP composite gels and films were measured for k1 spaceman at room temperature. Figure (4) represents the densities of all aqueous solutions increased by the increasing of the ration of SiO_2NPs, because of the gels formed across linked among the molecules of PEO/PMMA/PVP and ZrO_2NPs that occupied the spaces between PEO/PMMA/PVP molecules, furthermore, the density increased with increasing of doping materials [9].

The US transmittance waves (T) have been computed by [10]:

$$T = I / Io \text{ ………………. } (1)$$

From Figure (6) we notice that, the USW velocity of (PEO/PMMA/PVP) decreased with the increasing of frequencies, and computed by [11]:

$$V = X / t \text{ …………..…… } (2)$$

This behavior return to structural relaxation that happens in the associated PEO/PMMA/PVP and ZrO_2NPs composite with various frequency values. A collide at rest has internal characteristics similar to solid, but when the waves propagate resulted different periodic causes molecules flow between spaces in the lattice during compressing and finally return to the original position. The USW velocity has been directly proportion with ZrO_2NPs added, but inversely proportional with frequency, because USW causes various physical interaction between PEO/PMMA/PVP and ZrO_2 NPs molecules, lead to increase the velocity, but the increasing of frequencies led to decrease the velocities [12]. Figures (6 and 7) show that, the relaxation time and relaxation amplitude also decrease against the frequencies according to theoretical equation [13]:

$$D = \alpha / f^2 \text{ ……………… } (3)$$

The increasing of PEO/PMMA/PVP doped with ZrO_2NPs chains led to increase the fraction between the composition layers that examined by moment of inertia [14].

The compressibility of PEO/PMMA/PVP doped with ZrO_2NPs were theoretically calculated by Laplacian equation [15], so the values increased by the increasing of frequency:

$$\beta = (\rho\ v^2)^{-1} \quad \text{……………..} \quad (4)$$

The Young modulus (K) was calculated by [16]:

$$K = \rho\ v^2 \quad \text{………………} \quad (5)$$

The results in Figure (8) show that the compressibility of PEO/PMMA/PVP were increased with the increasing of frequencies, this is because the propagation of USW made a random polymer chain conformation, in addition of the USW make a compression lead to reduce the elasticity of composition [17]. Furthermore, the velocity of USW is inversely proportional with compressibility. Figure (9) represent, the bulk modulus decreased with increasing of frequencies. The specific acoustic impedance (SAI), Figure (10) is also showed the composite has been decreased with increasing of frequency depending on theoretical equation [18]:

$$Z = \rho\ v \quad \text{………………..} \quad (6)$$

The density is very small as compare with velocity. The USW absorption coefficient calculated by the law of Lambert – Beer [19]:

$$A/A_o = e\ (-\ \alpha\ x) \quad \text{……..} \quad (7)$$

Figure (11) also inversely proportional with the frequencies, and this is because USW absorption coefficient depends on the frequencies value [20-23].

6. Conclusions

PEO/PMMA/PVP/ZrO_2NPs novel composite films were successfully prepared by casting method at thickness of (80-95) μm. The OM and SEM images show a strong diffusion of NPs in the mixtures, furthermore FTIR peaks refers to good interactions between the raw material and NPs. The results also refer to NCs suitable for using in the many coating purposes. The new composite films can be used as resistant materials versus environment. The NCs havehuman skin harmless property. The obtained results show that most of physical properties were affected by the increasing of ZrO_2NPs. Most of the results were enhanced after doping and presented higher values than original. When, the ZrO_2NPs increased the density, viscosity, USW velocity and USW absorption coefficient of the polymer doped ZrO_2NPs were also increased. In addition, ZrO_2NPs led to increase the compressibility of USW and thus became more tolerable to environmental conditions and can be used in the modern external environment, and many industrial applications. In this study, it was found that the best frequency value is (25 MHz), which showed the best mechanical USW results. The mechanical properties enhanced after frequency varied such as, ultrasonic velocity and bulk modules. The dielectric constant of the k1 spaceman was (85%) increased with increasing of applied load. As a result, USW sensors can be made from (PEO/PMMA/PVP)/ZrO_2 NCs film.

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Figures

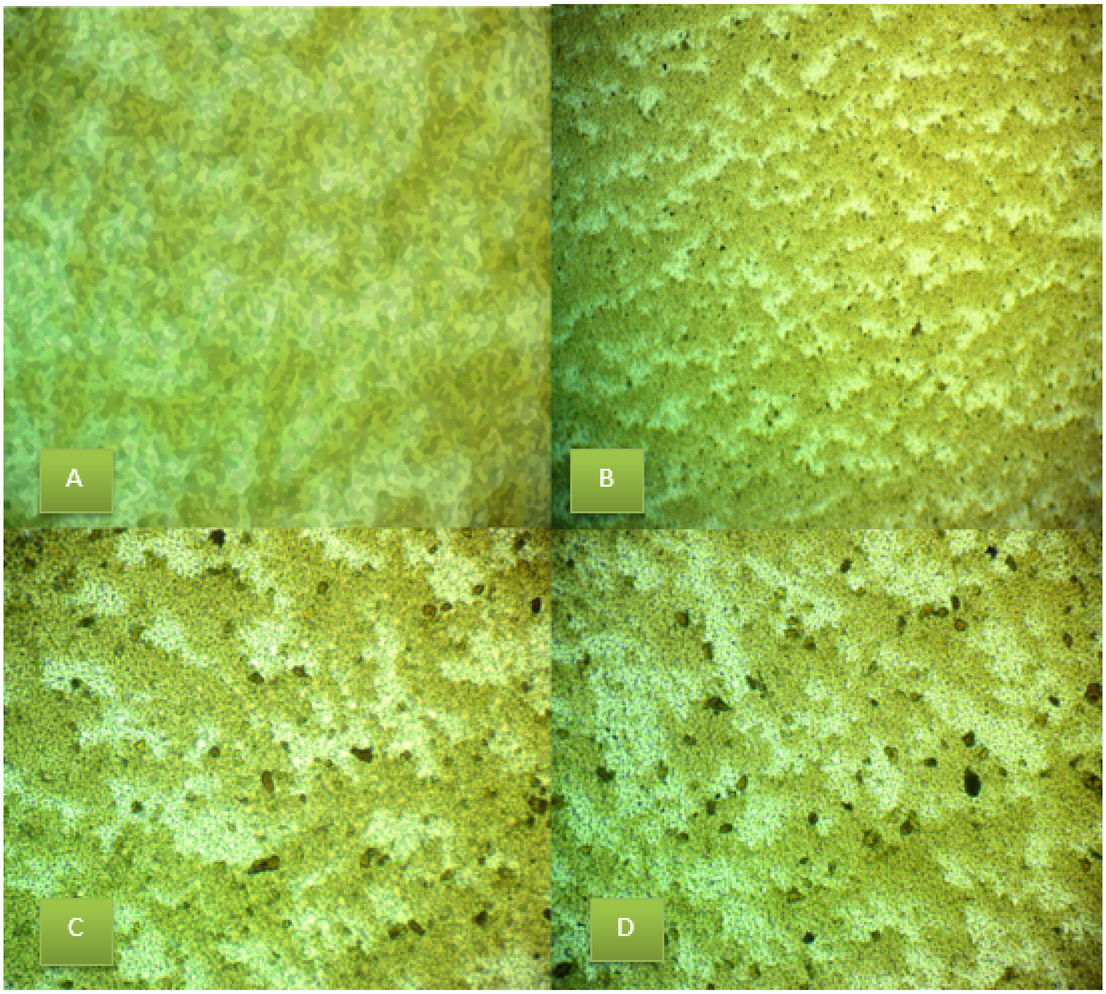

Figure 1

Photomicrographs (100X) of NCs: A) k0, B) k1, C) k2 and D) k3.

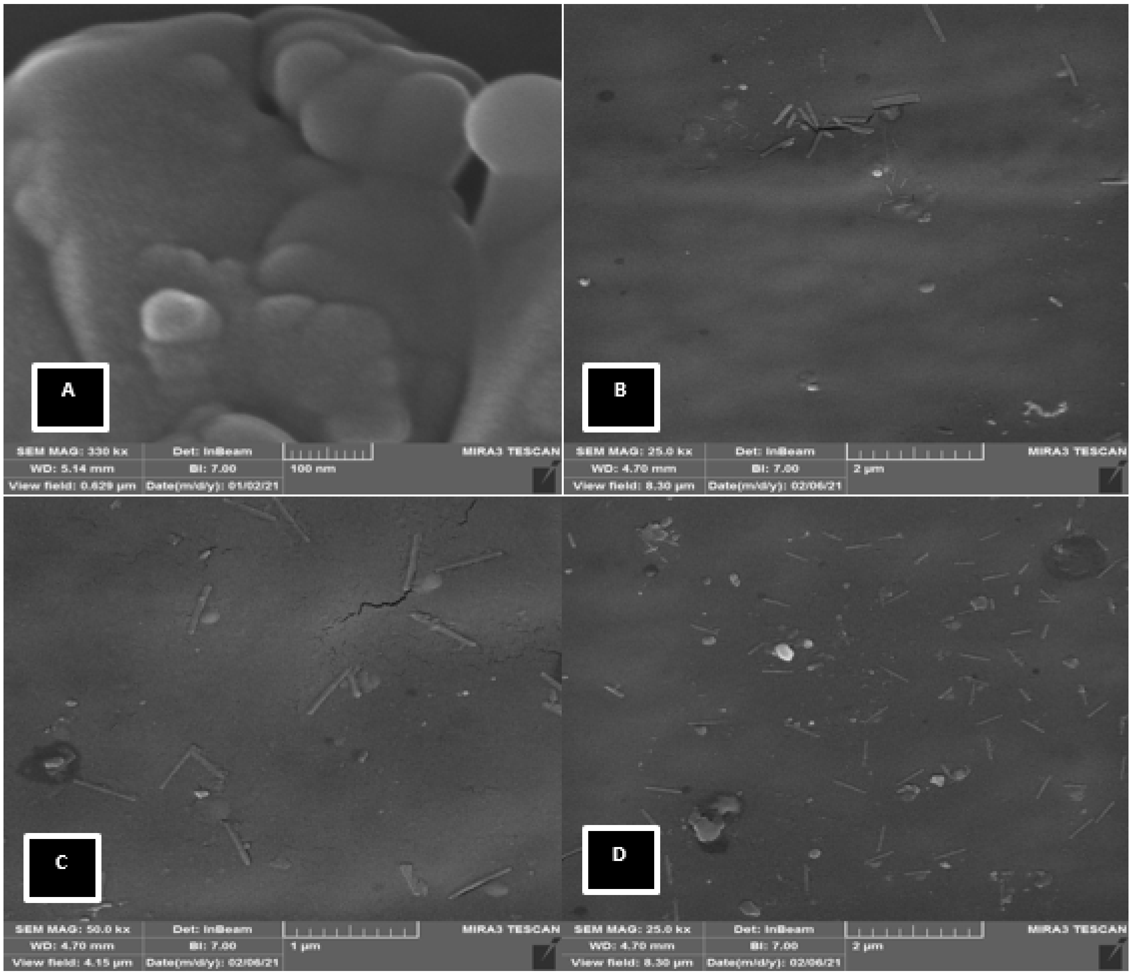

Figure 2

SEM images of: A) k0, B) k1, C) k2 and D) k3.

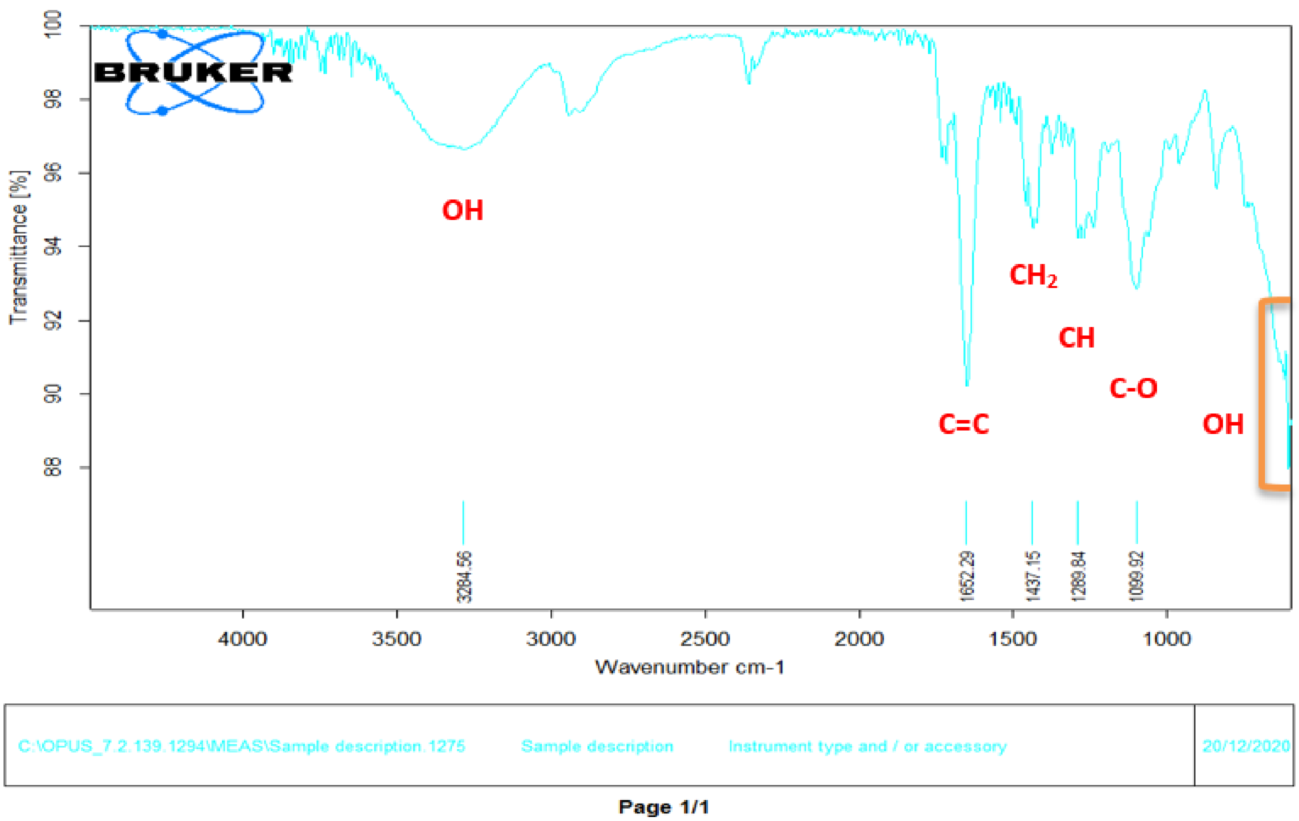

Figure 3

FTIR spectrum of k1 specimen.

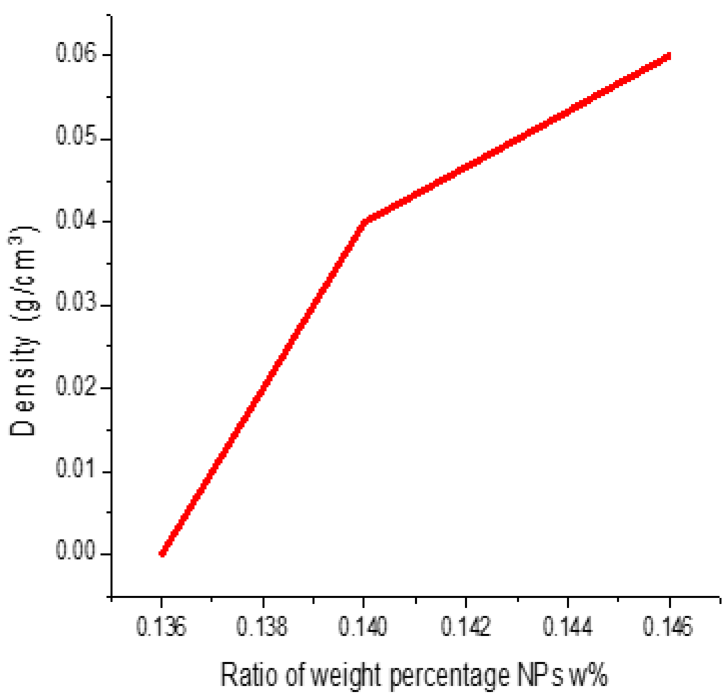

Figure 4

Density vs. SiO2NPs%

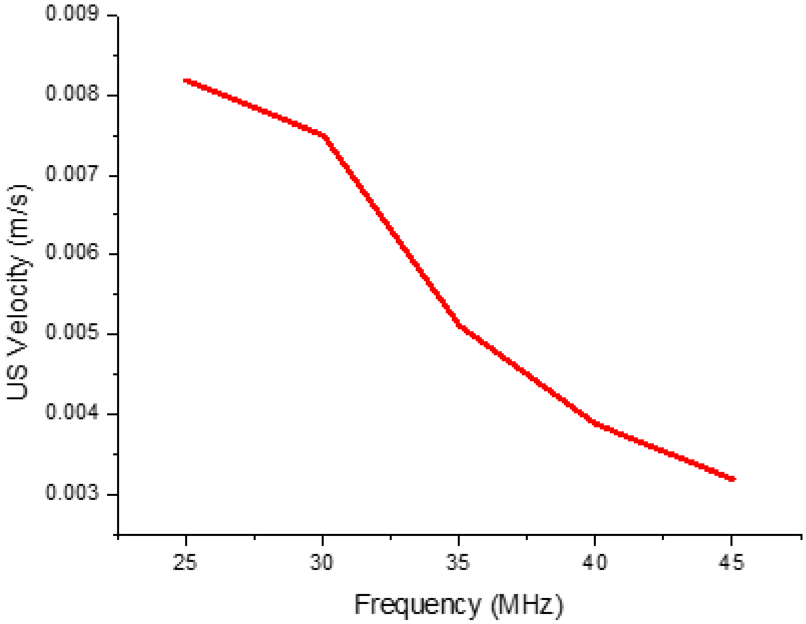

Figure 5

USW velocity vs. frequency

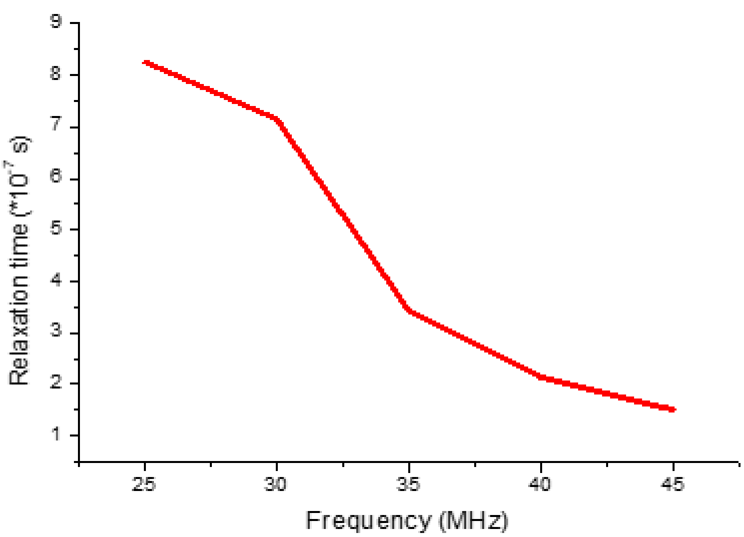

Figure 6

Relaxation time vs. frequency

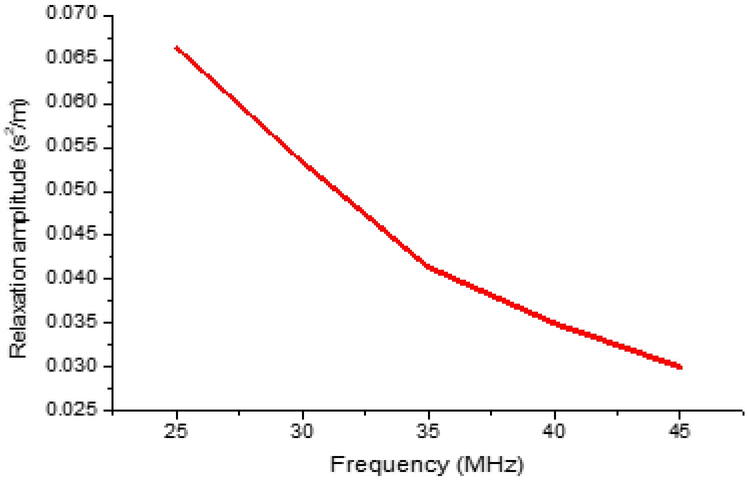

Figure 7

Relaxation amplitude vs. frequency

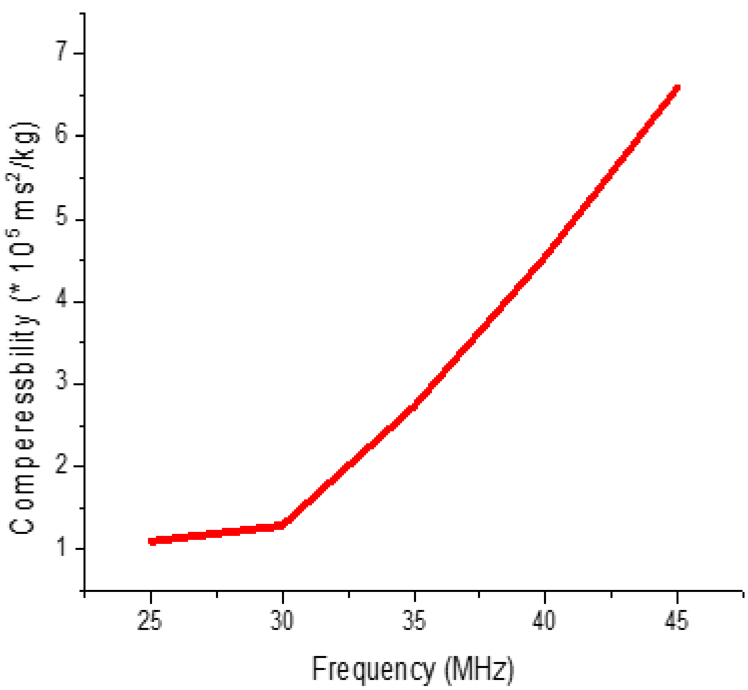

Figure 8

Compressibility vs. frequency

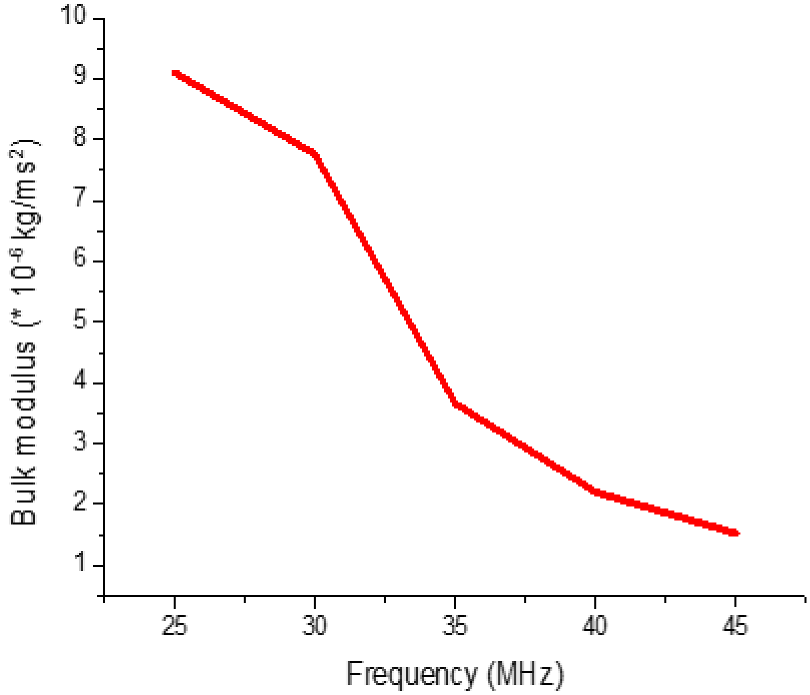

Figure 9

Bulk modulus vs. frequency

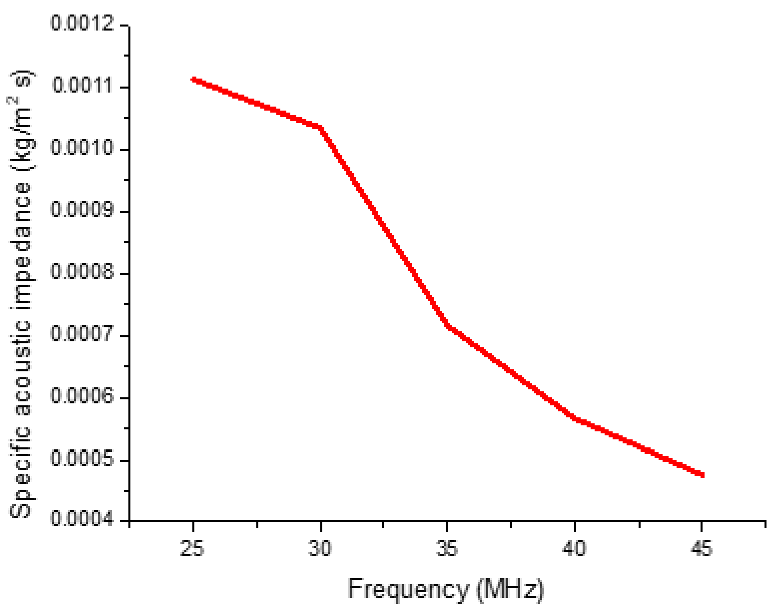

Figure 10

SAI vs. frequency

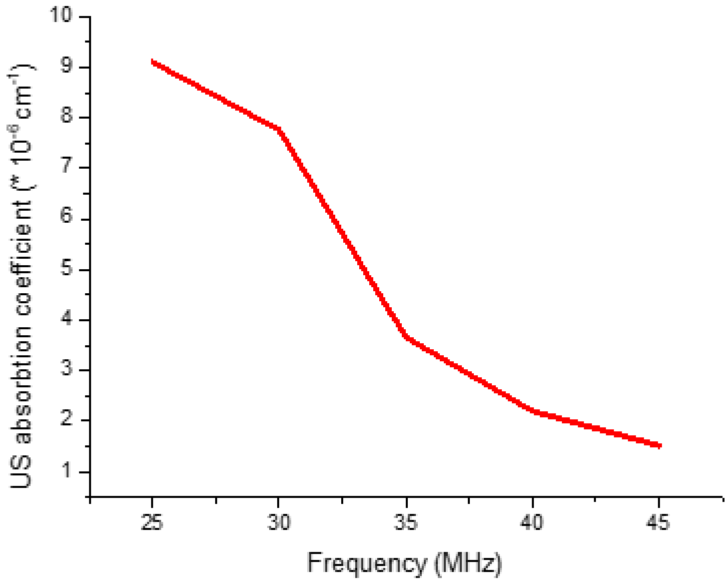

Figure 11

US absorption coefficient vs. frequency

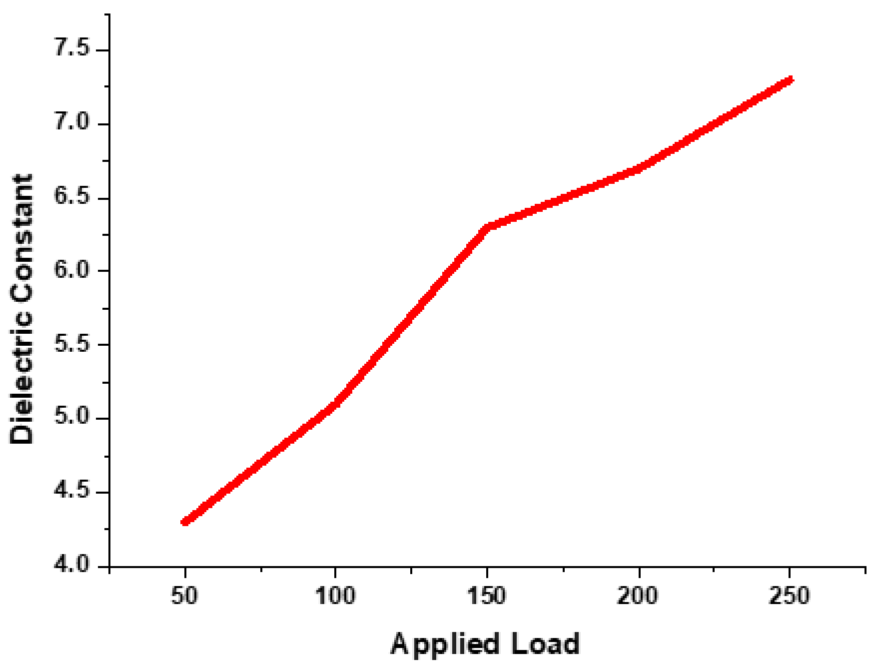

Figure 12

Dielectric constant vs. applied load of k2 spaceman.

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