Preparation of Metal Organic Compounds containing Schiff Bases and Study Some of its Mechanical Properties

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Abstract

This research included preparation of complexes for Ni ion chloride(II) Cr ion chloride(III) and aqueous iron chloride (III) with Schiff bases ligand (N, N) di methyl of the amino Benzaldhyde. The prepared complexes were characterized by IR, UV and visible spectra. Also the mechanical properties by technique of ultrasonic waves velocity at 40 KHz frequency were studied include the ultrasonic waves velocity, absorption coefficient of ultrasonic waves, relaxation time, relaxation amplitude, specific acoustic impedance compressibility, and bulk modules. The Results showed that all properties decrease with increase the ultrasonic waves velocity except the specific acoustic impedance and bulk modules which directly proportional to the velocity and all the results were compared with with those for similar compounds.
Keywords: Metal Organic Compounds, Schiff Bases, Mechanical Properties.

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الخلاصة

تضمن هذا البحث تحضير معقدات لايون النيكل الثنائي التكافؤ(II) والكروم المائي الثلاثي التكافؤ (III) وايون الحديد الثلاثي التكافؤ (III) مع ليكاند (المادة الحرة) قاعدة شف [(N،N–ثنائي مثيل أمينو بنزلديهايد)] . تم تحليل المعقدات المحضرة بوساطة طيف الأشعة تحت الحمراء وطيفي الأشعة فوق البنفسجية والمرئية , كما اشتمل البحث على دراسة الخصائص الميكانيكية بوساطة تقنية الموجات فوق الصوتية وبتردد (40)KHz, فقد تم قياس سرعة هذه الموجات ومعامل الامتصاص وزمن وسرعة الاسترخاء والممانعة الصوتية النوعية والانضغاطية ومعامل المرونة وأظهرت النتائج إن جميع هذه الخصائص تتناقص مع زيادة السرعة ماعدا الممانعة الصوتية ومعامل المرونة فإنها تزداد بزيادة السرعة وقد قورنت جميع النتائج مع مركبات شبيهة .

الكلمات المفتاحية: المركبات العضوية الفلزية, قواعد شف, الخصائص الميكانيكية.

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Introduction

Strictly speaking Schiff bases are compounds having a formula RR'C=NR'' where R is an aryl group, R' is a hydrogen atom and R'' is either an alkyl or aryl group. However, usually compounds where R'' is an alkyl or aryl group and R' is an alkyl or aromatic group are also counted as Schiff bases class is very versatile as compounds can have a variety of different substituents and they can be unbridged or N, N bridged. Most commonly Schiff bases have NO or N_2O_2 -donor atoms but the oxygen atoms can be replaced by Sulphur, Nitrogen or Selenium atoms. In this study, attention was paid to the most common Schiff bases, especially to salen and salophen Complexes. El-ajaily studied the antibacterial activities of the Schiff base derived from the salicylaldehyde and histidine and its Mn(II) ،Co(II) 'Ni(II) ' Cu(II) and Cd(II)complexes on some pathogenic bacteria .The divalent metal ions of Co(II)، Ni(II)or Zn(II) have the formula of Ml_2x_2 and Ml_2 in there complexes. The Mechanical properties were studies for hydroxy ethyl cellulose polymer in which they repotted by (Al-Bermany, 1995) and all the Results showed that all properties increased with the increase of the concentration except the Compressibility which was decreased with increase the concentration

(Al-Bermany, 1995). The present work aims to synthesis sample from through of correction Schiff bases compounds and study some of mechanical properties of Schiff bases complexes.

Theoretical Part:

Measurements and Calculation

The Ultrasonic velocity (V) was measured using the pulse ultrasonic technique of sender- receiver type SV-DH-7A, SVX-7 with (0-50 kHz) variable frequency, the metal vibrator was coated with oil and kept in contact with the wall of the glass tank that containing the test sample the receiver quartz crystal was mounted on a digital vernier of slow motion. The receiver crystal could be displaced in parallel to the sender through 10 Cm. The sender and receiver pulses were displaced on two traces of cathode ray oscillograph. Theoretical values such as absorption coefficient α of ultrasonic waves, relaxation time τ, relaxation amplitude D, compressibility β ' specific acoustic impedance Z and bulk modulus K for the polymer solutions have been calculated using the following equations (Blitz; 1967; Blitz, 1967; Hassun,1990;Hassun, *et al.,* 1990; Al-Bermany,2009; Al-Berman, 2010) The absorption coefficient α had been calculated by:

$$\alpha = \frac{-\ln A/A_0}{X} \qquad \text{-----------------------(1)}$$

Where A and A_0 are ultrasonic wave amplitude and initial wave amplitude, respectively and X is the crystal moving distance

The relaxation time τ had been calculated as :

$$\tau = \frac{4\eta}{3\rho V^3} \qquad \text{-----------------------------(2)}$$

where η is the viscosity of solution, ρ the density, V velocity of ultrasonic waves

The relaxation amplitude *D* had been calculated by the following equation

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

where f is the frequency of ultrasonic waves

The specific acoustic impedance *Z* given by :

$$Z = \rho V \qquad \text{----------------------------(4)}$$

The compressibility *β* and the bulk modulus K are computed from equations (5) and (6) , respectively .

$$\beta = (\rho V^2)^{-1} \qquad \text{--------------------(5)}$$

$$K = \beta^{-1} = \rho V^2 \qquad \text{-----------------(6)}$$

Experimental Part

Synthesis of Schiff Base Ligand

The method of preparation was as follows:

The *4*-amino antipyrine (5 g, 0.014 mol) dissolved with 4-dimethylamino benzaldehyde (3.6 g, 0.024 mol) in ethanol (40 ml). Five drops of glacial acetic acid added to the solution and the mixture was refluxed for (1.5hr). The Schiff base compound was isolated after the volume of the mixture was reduced to half using rotary evaporation and the obtained product was collected by filtration, washed several times with ethanol and recrystilized from absolute ethanol the melting point of the yellow crystals found to be (219 ^{o}C). The yield was (99.99 %).
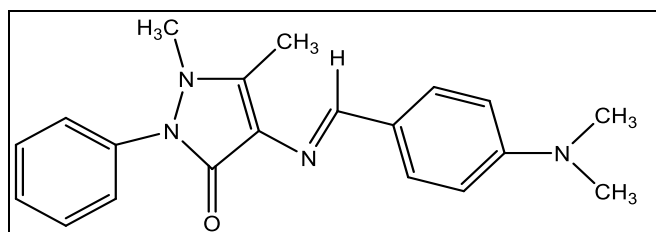

Fig: 1 Structural formula of the ligand (free material)

Preparation of Schiff Base Complexes:

The prepared Schiff base (3.34 gm, 0.01mol) in 30 ml ethanol have been mixed with metal salts [1.29 g $NiCl_2$ (0.01 mol), $CrCl_3.6H_2O$(2.66 g), $FeCl_3$ (0.8 g)] respectively in the same amount and refluxed for two hours. The resulted complexes were collected by filtration and then washed several times with ethanol, dried and stored. All the prepared compounds have been characterized using infrared (IR) and ultraviolet and visible (UV-Vis) spectra.

Results and Discussion

It is clear from comparing the spectrum of the (IR) of this complex with ligand spectrum they observed differ, the new packages were not already present in the spectrum of ligand while suffered other packages from the obvious changes in shape, intensity and location. The reason for this is to get the consistency between the metal ions and free material. Has been benefiting from the results of measurements of UV and (UV-Vis) spectra to see the transfer of electronic to these complexes is attributed these packages to the state of excitement topical for benzene ring because for absorption of the groups (C = N), (C = C), (CH), (MN) resulting in general for transitions (n-π*), (π-π*) is expected to be packaged absorption at wavelengths higher back for the transition (n-π*) of the azomethine group effective (C=N) donor of the electrons. Available as an electronic pair not bounded is the nitrogen atom in which can be shared in the process of moving mail. Absorption coefficient of ultrasonic was calculated by measuring the distance and amplitude according to equation (1) to get the values the process of absorption coefficient , as shown in Figure (2) the increasing of ultrasonic velocity is are large, which makes the particles due to the situation stable duration is shorter, due to structural relaxation happening between the molecules of the compound is the one who will be responsible for the decrease in the absorption of ultrasonic, as well as a process of cross linking, which leads to random distribution of particles of the compound and the solvent and this is confirmed by the increase in the values of the ultrasonic velocity, so less than the values of coefficient absorption as a result of random distribution, either at the velocities the few we find absorption coefficient takes high values for the material

free (a) compared complex (c) and complex (d) where we find value at these velocities is not large, and this is due to break the bonds because of few of cross linking in the Free material (a) [Al-Uqaily, 2004]. And thus acquire higher energy particles and therefore has the ability to return to its original status as a shorter duration of time (Hassun, 1990; Hassun*et al.,* 1990). Can be calculated the values of the relaxation time using the molecular value measured for each of the density and viscosity, velocity, according to the relationship (2), and Figure (3) shows the relationship between the relaxation time, velocity, show the decrease in the relaxation time with increasing velocity to get the cross linking between the moleculars compound that restricts the movement of these moleculars in their positions, leading to the decrease in the values of the relaxation time of any decrease in the time required to re-molecule raised to its original status, as the relaxation time is less in the compound (a) The reason is when the passage of ultrasound in the solution lead to break bonds for complexes both (c, d) and as a result of broken bonds for the purpose of stability and re-entry into their original positions require a period of relaxation is greater than its predecessor in the molecules of the compound ((a. (Figure 4) shows the relationship between the relaxation amplitude and velocity, as seen from the Figure that the relaxation amplitude decrease with increase velocity and the reason is due to the small distance traveled by the molecule in the process of arousal, because of the determination of the inertia of the large molecular is small (Al-Nasraoui, 1998; Al-Nasraoui, 1998) and we show that the moleculars at velocities few took the character of cross linking after a break bonds and increase the size of the moleculars and then increased moment of inertia, which leads in turn to increase the relaxation amplitude and in particular a complex chromium as it takes high values for the relaxation amplitude at low velocities and due to the presence of water molecules associated with the bonds of hydrogen, which are themselves the bonds are weak as quickly broken when the pass of ultrasonic to the solution of the complex referred to, and this action is reversed when increasing velocity, as well as the relaxation amplitude is directly proportional with the absorption coefficient as shown in relation (3), and these results are correspond with the results of researchers (Hassun ,1990, Al-Bermany ,2003; Al-Bermany *et al.,* 2003) and when the frequency constant (40) KHz for all models is therefore expected to increase the relaxation amplitude of the molecules of the solution when increasing the absorption coefficient. Figure (5) shows the relationship between the Specific acoustic impedance and velocity, as seen from the figure when you increase ultrasonic velocity increases specific acoustic impedance form linearly and the reason goes back to the increase the number of molecular due to increased velocity, which in turn leads to increased density of the medium as the passage of ultrasonic within the solution lead to a compression and rarefaction, where there is the breaking of the bonds of weak and the formation of free roots in chains (material free and complexes) that are inherently unstable and that must be interdependence again to get the state of stability and thus configure the phenomenon of cross linking in the solution, which lead to increased specific acoustic impedance expressed by the solution as a result of the case of overlapping cross and increase the medium density, thus increasing the specific acoustic impedance and figure (6) shows the changing values of compressibility with velocity, as it shows Figure decrease the values of compressibility with increasing velocity, and the reason for this is due to the convergence of moleculars the solution led to the occupancy of space in the solution, which in turn led to a decrease of compressibility (Bajpai,2003; Bajpai, *et al.* 2003]. This is consistent with what obtained by the researchers (Suad, 1993, Hassun, 1990; Abdul-Majid, 1993). Since the compressibility is inversely proportional with velocity as described in equation (6), they lead to decreased compressibility where we note in free

material of compressibility value at the highest velocity first because of the lack of overlap between the particles of medium which makes the compression takes a high value on the opposite than in the case of complex (c) and (d) except that the compressibility decreases quickly because of the complexity of the solution molecules led to the decrease in compressibility (Musa, 2004) (Figure 7) show that increases bulk modulus with increasing velocity, as note from Figure increasing values and the reason for this through the relationship (7) bulk modulus on the ultrasonic velocity, mainly so it's expected behavior of bulk modulus the same behavior of ultrasonic velocity , this result is consistent with the findings of researchers (Al-Bermany,1995, Khaleda , 2001; Al-Bermany, 1995] where the note in iron complex of that the bulk modulus record the highest values him at velocities large because of the high density of this complex compared complexes other and followed the free material (a) and then complexes chromium ، nickel, iron (b, c, d) respectively (Al-Amery, 2003).
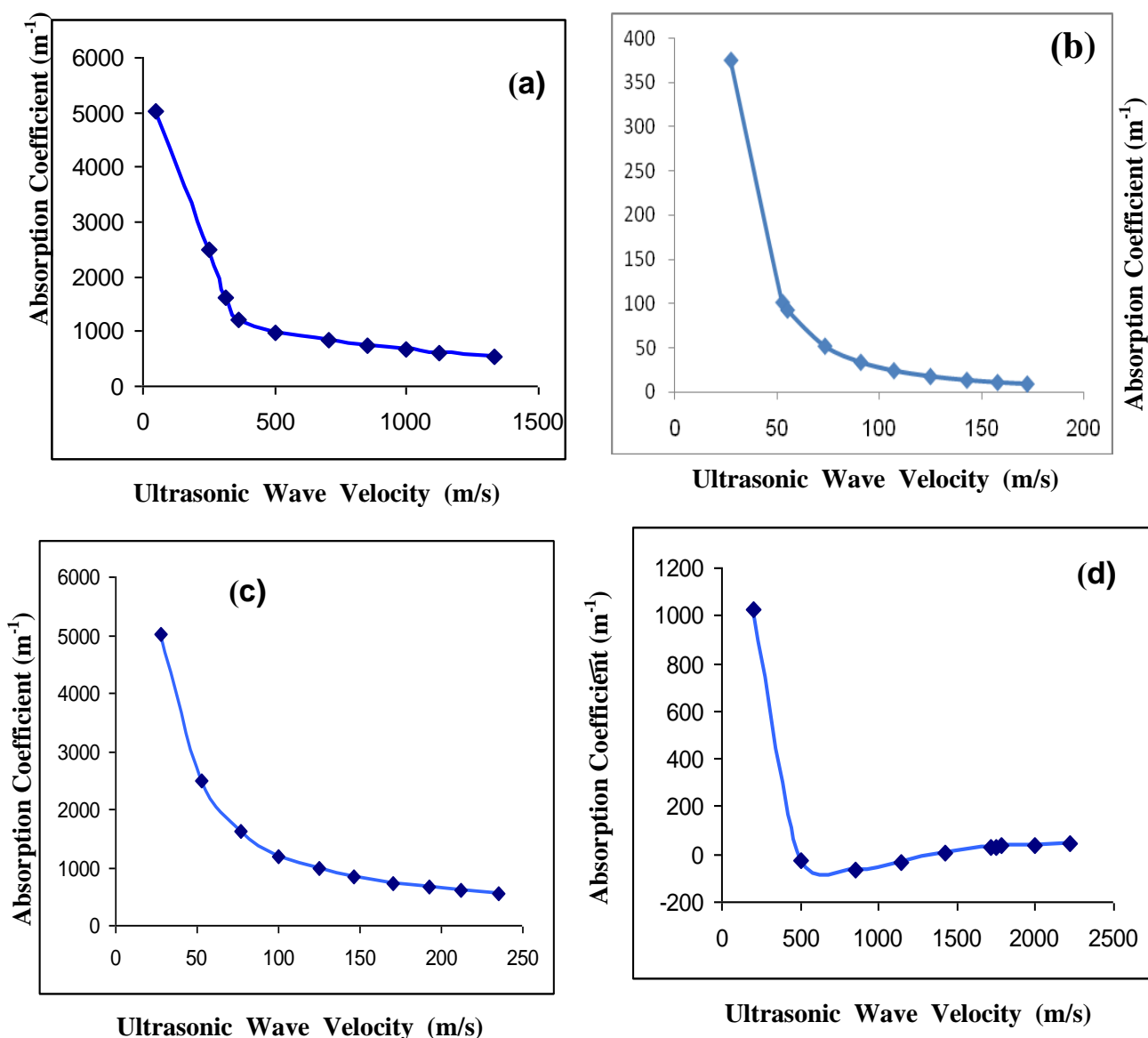

Fig.2 the variation Absorption Coefficient Vs ultrasonic velocity of prepared compounds

    (a): ligand (free material)
    (b) nickel bivalent (II) chloride
    (c): chromium aqueous trivalent (III) chloride
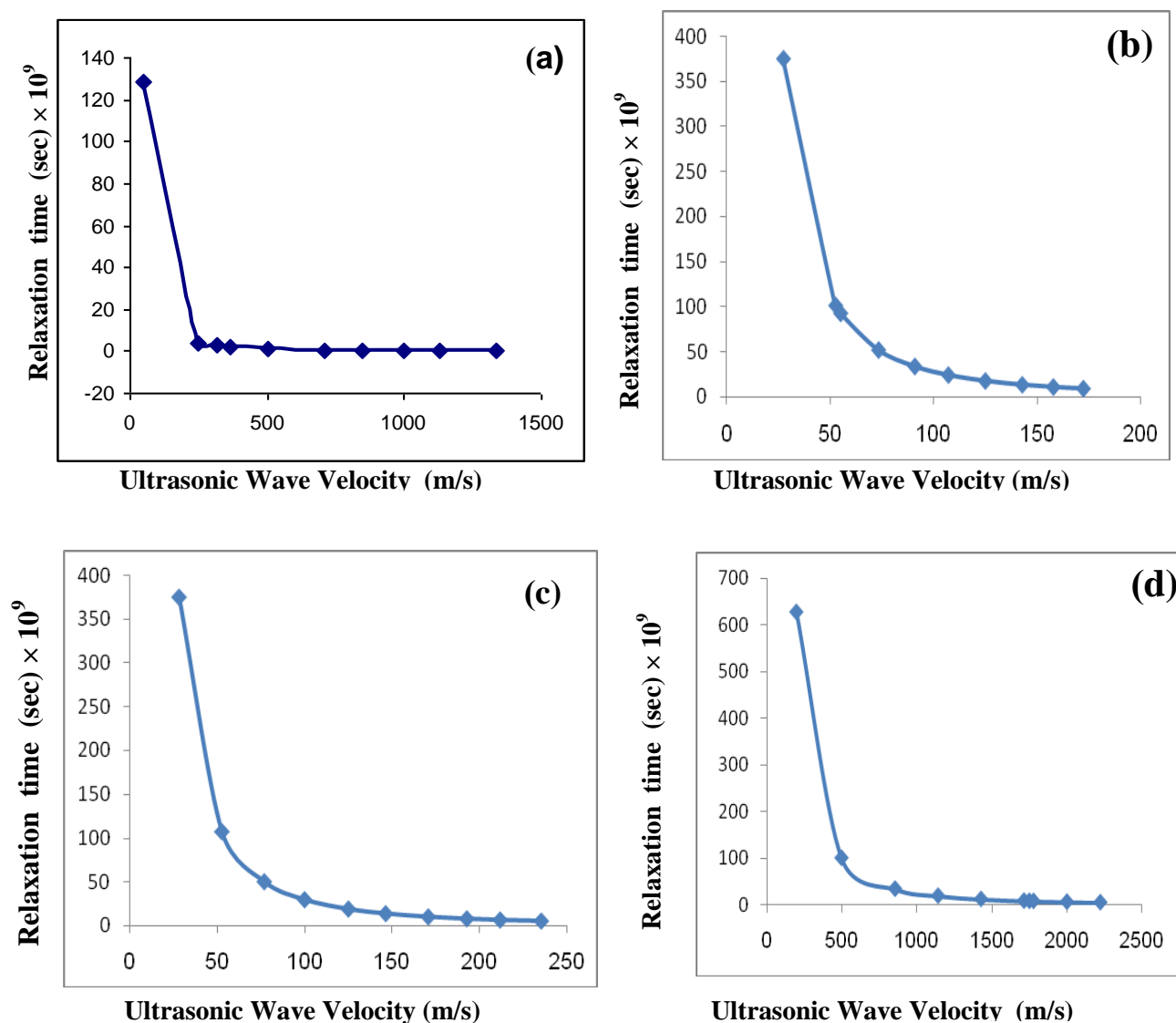   (d): iron trivalent (III) chloride

Fig.3 The variation relaxation time Vs ultrasonic velocity of prepared compounds
(a): ligand (free material)
(b) nickel bivalent (II) chloride
(c): chromium aqueous trivalent (III) chloride
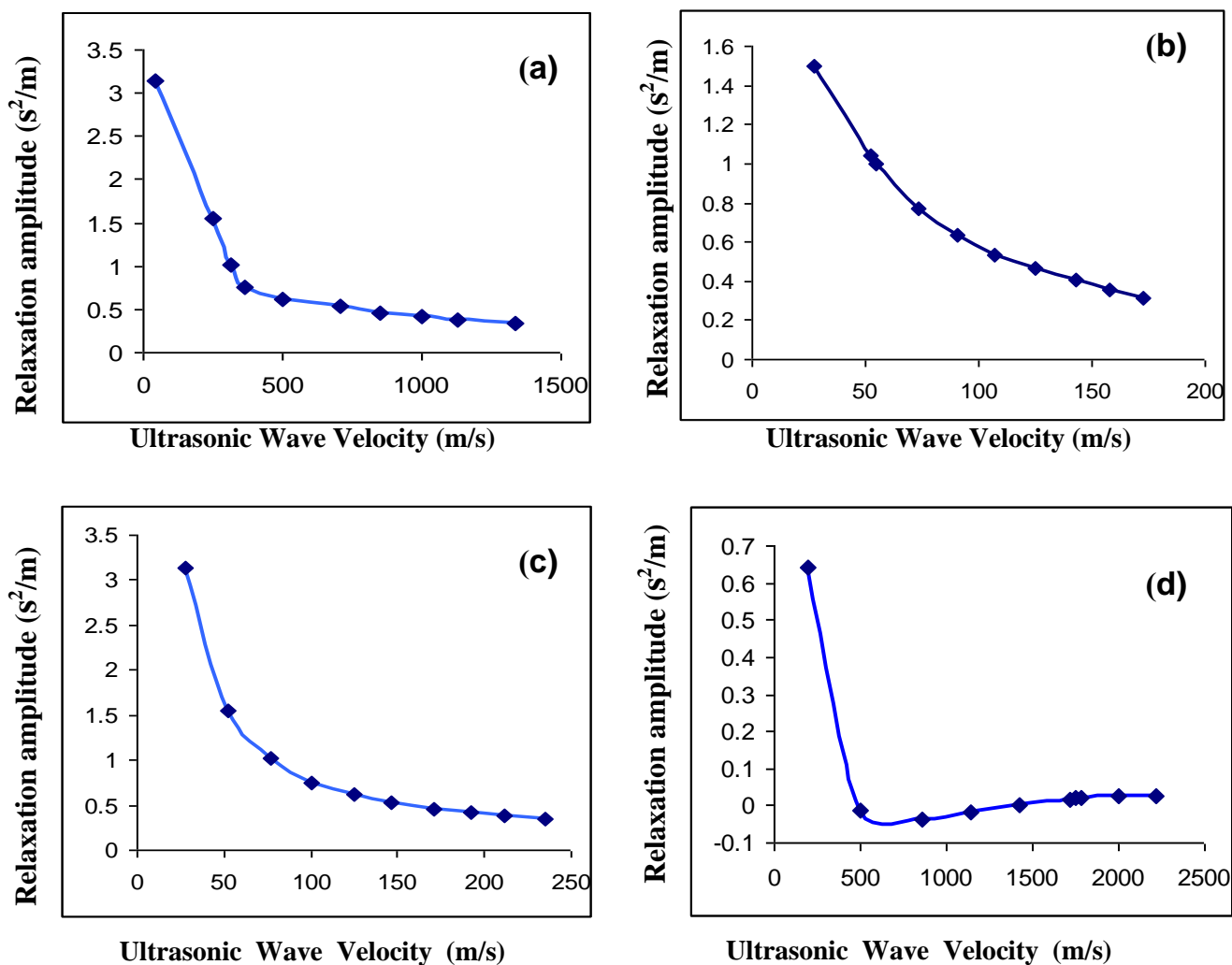(d): iron trivalent (III) chloride

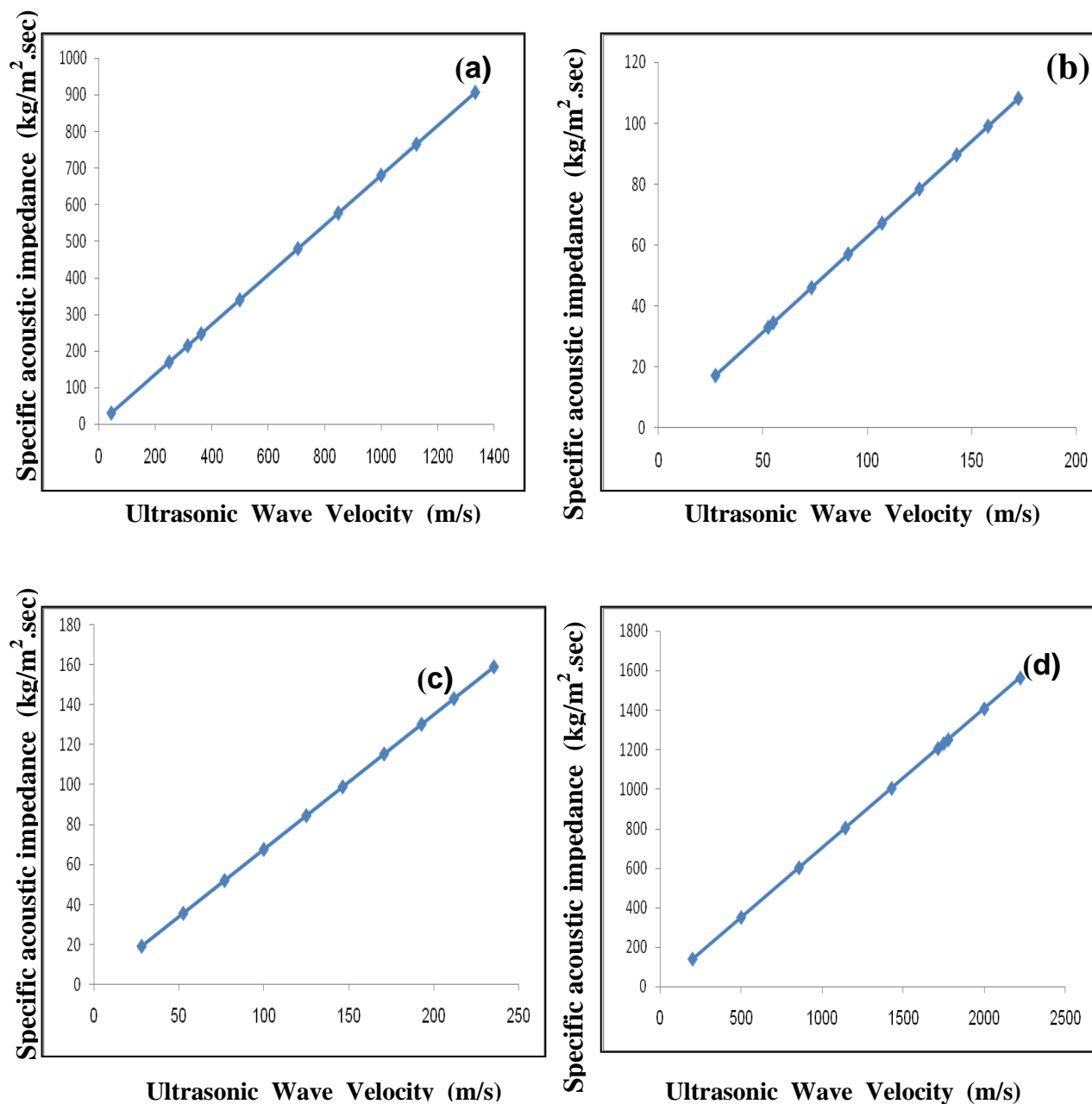**Fig.4 The variation relaxation amplitude Vs ultrasonic velocity of prepared compounds**

(a): ligand (free material)
(b) nickel bivalent (II) chloride
(c): chromium aqueous trivalent (III) chloride
(d): iron trivalent (III) chloride

Fig.5 The variation specific acoustic impedance Vs ultrasonic velocity of prepared compounds

> (a): ligand (free material)
> (b) nickel bivalent (II) chloride
> (c): chromium aqueous trivalent (III) chloride
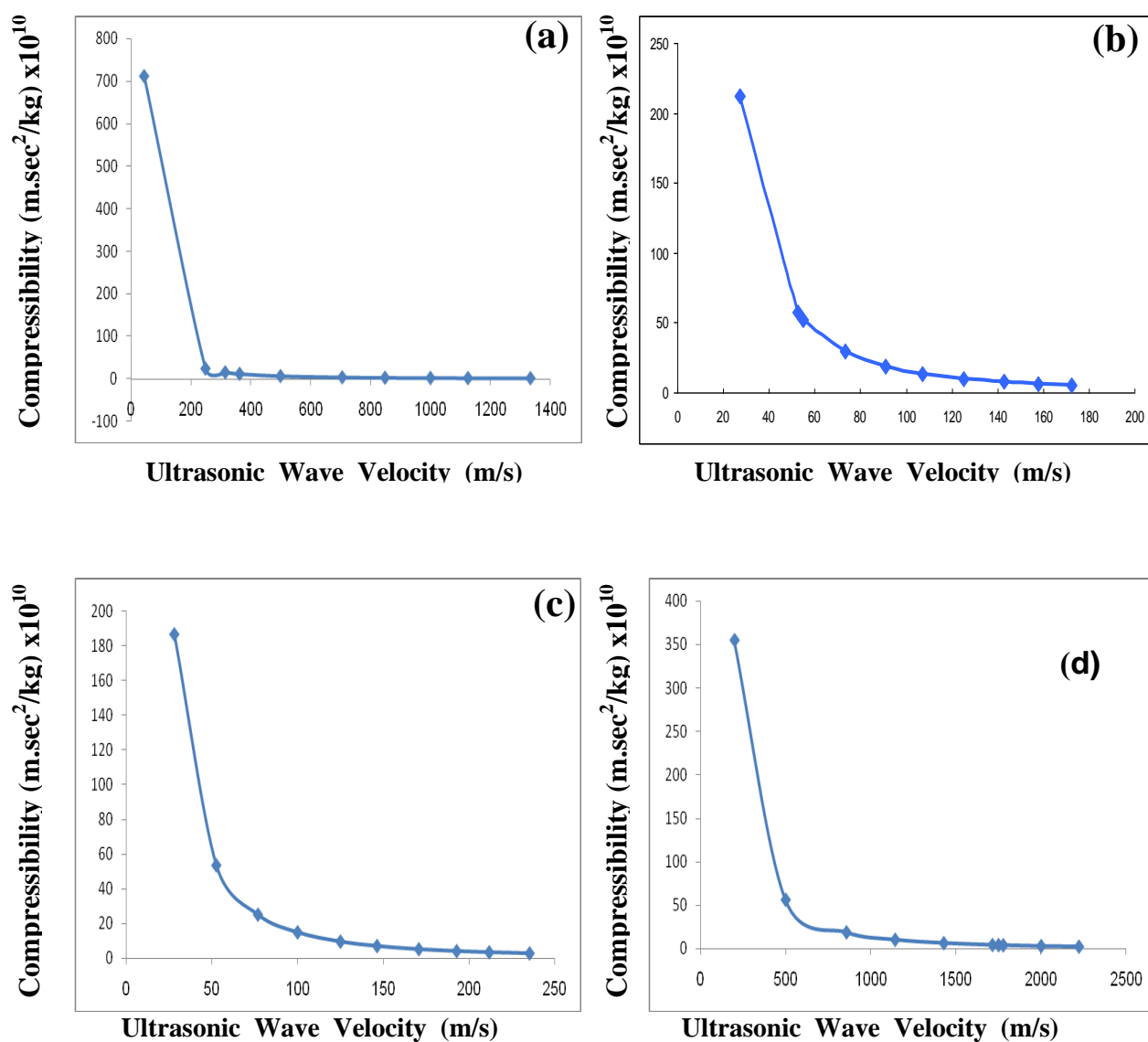> (d): iron trivalent (III) chloride

Fig.6 the variation Compressibility Vs ultrasonic velocity of prepared compounds

     (a): ligand (free material)
     (b) nickel bivalent (II) chloride
     (c): chromium aqueous trivalent (III) chloride
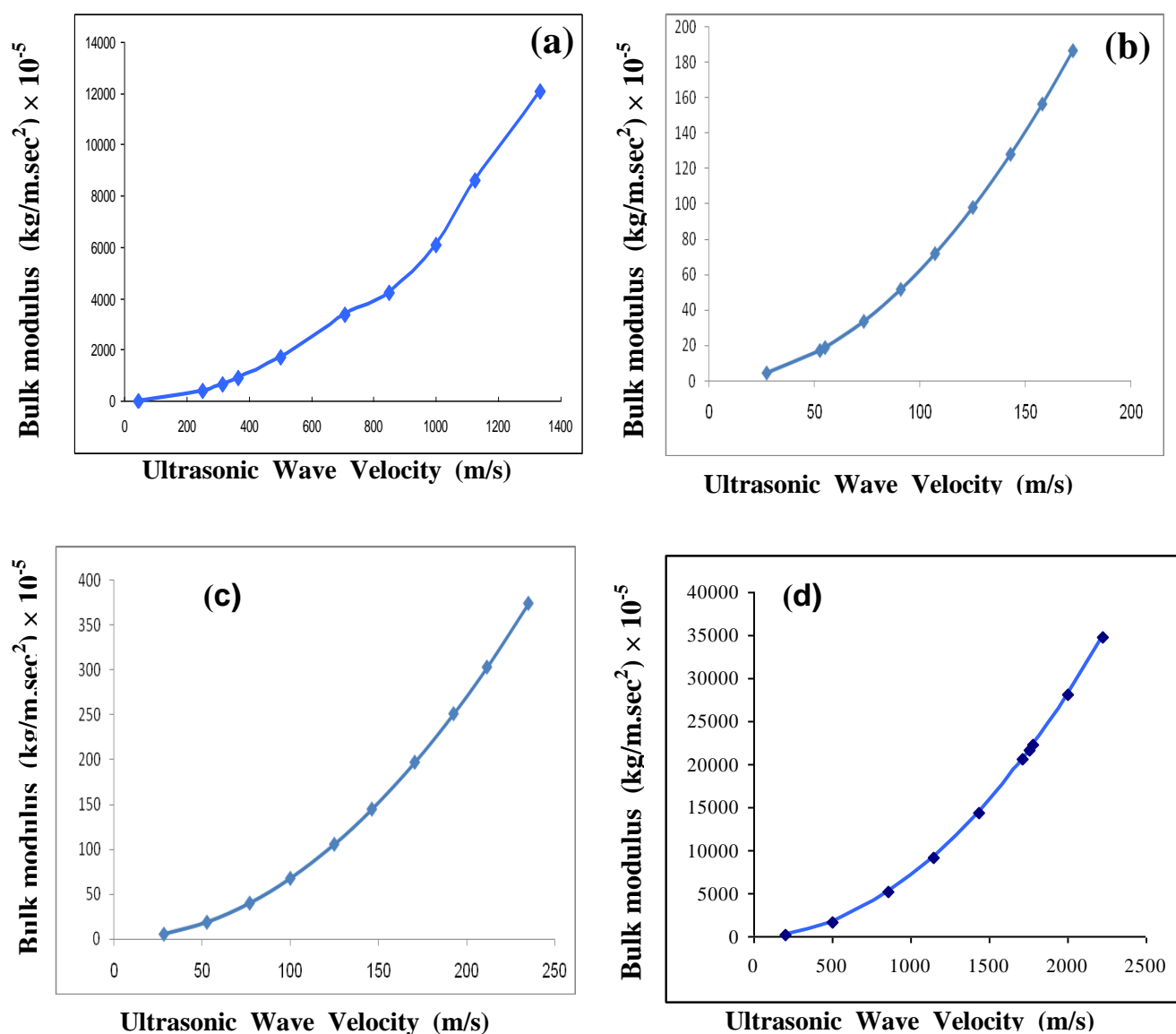   (d): iron trivalent (III) chloride

Fig.7 The variation bulk modulus Vs ultrasonic velocity of prepared compounds

(a): ligand (free material)
(b) nickel bivalent (II) chloride
(c): chromium aqueous trivalent (III) chloride
(d): iron trivalent (III) chloride

Conclusions

1. Ease of preparation of this type of ligand accompanied by some of the difficulties in purifying prepared compounds

2. Characterized metal complexes shows not affected by the circumstances of the light, humidity, and air, which refers to the perceived stability, in addition to its relatively high degrees of melting, which gives further evidence of the extent of stability. 3. The process of absorption and attenuation of ultrasonic energy are very dependent on both the length of the chain (free material and prepared complexes under study) and the velocity of these waves with respect to its mechanical properties, while with respect to the optical properties were highly dependent on the length of the chain (free material and prepared complexes under study) and the concentration of the solution.

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