

**Republic of Iraq  
AL-Nahrain University  
College of Science**



**A STUDY OF THE EFFECT OF ELECTROLYTE SOLUTION  
ON THE PROPERTIES OF ELECTRICAL INSULATOR  
PORCELAIN FORMED USING LOCAL MATERIAL**

**A Thesis**

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## *Abstract*

This study was carried out to prepare a porcelain samples, which can be used as an electrical insulator. The raw materials used for the preparation are kaolin Duakhla, Arudhma Sand glass and Potash feldspar with weight percentage (45%, 30%, 25%) respectively. A chemical treatment was performed on kaolin Duakhla, Arudhma Sand glass to remove all the impurities. The effect of the washing by HCl acid (chemical treatment) was examined by IR Tech.

The study divided into seven groups. Relating to the concentration of electrolyte solution which is consisting of ( $\text{NaCO}_3$  &  $\text{NaSiO}_3$ ) as a ratio (2:1), and for fixed percentage of mineralizer (2%ZnO). The concentrations of electrolyte which were added to these groups are (1%, 0.7%, 0.5%, 0.2%, and 0.1%). Samples of each group have been prepared, and then each sample was pressed, then after that these samples are dried and sintered for different temperatures (1250, 1300, 1350 °C). Dielectric measurement (Dielectric Constant& Dielectric loss index) were carried out for these pressed samples, also Physical properties “Thermal conductivity” and mechanical properties “Compression strength” were measured for these samples. The results of these measurements for samples sintered at 1350°C are found to have a stable change at the range (0.1–0.2%) of concentration of electrolyte solution. While there is a change in the results of thermal conductivity in the concentration range (0.2–0.5%) of electrolyte solution. And the values of compression strength is found to be high at 0.5% concentration of electrolyte solution. Therefore we should select the best concentration of electrolyte solution, to get the optimum properties which would agree with practical results and the standard properties for the attentive in other countries of the world. Then, can regarde the (0.2%) concentration of electrolyte solution is the optimum value that can be used in the industry of the electrical insulators.

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*List of symbols*

|                 |  |
|-----------------|--|
| A               | cross section area.  |
| $A_0$           | Original cross–section area of the sample.                     |
| C               | Capacitance of capacitor in medium.                            |
| da              | Parallel plate capacitor distance                              |
| ds              | Sample thickness.  |
| $D_d$           | Dielectric displacement.                                       |
| DLVO            | (Derjaguin–Landau–Verwey–Overbeek) theory.                     |
| $\frac{dT}{dx}$ | The temperature gradient through the conductivity              |
| e               | Emitted energy   |
| $\hat{e}_{ij}$  | Unit vector directed from i to j.                              |
| $\bar{E}_i$     | Microscopic field intensity at position i.                     |
| $\bar{E}$       | Electric field in the medium.                                  |
| $E_0$           | External electric field.                                       |
| EDL             | Electric double layer.   |
| F               | Normal forces.   |
| $F_i$           | Electrostatic force on charge $q_i$ at distance $r_i$ .        |
| I               | The ionic strength.  |
| IR              | Infrared radiation.  |
| K               | Thickness of electrical double layer is known<br>Debye length. |
| $K$             | The thermal conductivity.                                      |
| m               | Molar concentration  |
| $M_i$           | Percentage of solution before dilution.                        |

|                |   |
|----------------|---|
| $M_j$          | Percentage of solution after dilution.                  |
| $Na_2CO_3$     | Sodium Carbonate.                                       |
| $Na_2SiO_3$    | Sodium Silicate.  |
| P              | Polarization.   |
| PVA            | Polyvinyl Al–cohol.                                     |
| q              | Heat flux or heat flow.                                 |
| $q_i$          | Charge q in position i.                                 |
| $q_j$          | Charge q in position j.                                 |
| $r_d$          | Sample radius.  |
| $r_i$          | Distance at position i from origin.                     |
| $\vec{r}_{ij}$ | Vector directed from i to j                             |
| R              | The resistance of condenser filled with the dielectric. |
| $R_p$          | Resistance of the material.                             |
| $V_i$          | Volume of solution before dilution.                     |
| $V_j$          | Volume of solution after dilution.                      |
| XRD            | X – Ray Diffraction.                                    |
| Z              | Valance number.   |
| ZnO            | Zinc Oxide.   |
| ZP             | Zeta Potential .  |

*The Greek Symbols*

|               |  |
|---------------|--|
| $\epsilon$    | Dielectric Constant of the medium(permittivity)          |
| $\epsilon_0$  | Dielectric constant of vacuum(vacuum permittivity )      |
| $\epsilon'$   | Relative dielectric constant                             |
| $K''$         | Dielectric loss index( $K'' = \epsilon'' / \epsilon_0$ ) |
| $K^*$         | Complex dielectric( $k^* = k' - ik''$ )                  |
| $\tan \delta$ | Loss angle   |
| $\Phi_0$      | Microscopic potential                                    |
| $\rho_c$      | Charge density   |
| $\sigma$      | Compression strength                                     |
| $\delta$      | Stern Layer  |

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*Itab*  
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## *Supervisor's Certification*

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## *Chapter One*

### *Introduction & Theoretical Part*

#### **1.1 Introduction**

Ceramics display a variety of useful electrical and magnetic properties, and it is one of the important electrical insulators [1]. An insulator may be defined as a device for physically supporting and electrically isolating a conductor or other piece of electrical equipment [2]. In the last forty years a great deal of assessment has been carried out in the manufacturing of electrical insulators. Bauer and Dietz [3] discussed the future needs for porcelain insulators. Signer and Singer [4], Budnikov [5] classified the ceramic materials for the electrical industry and their general properties.

Dielectric materials possess a large energy gap between valence and conduction bands; these materials have a high resistivity. Two important applications for ceramics materials which include electrical insulators and capacitors. Insulators, simply used to prevent the charge transfer in an electric circuit, but used to store electric charge such as capacitors [1].

Ceramic insulators (more commonly, porcelain insulator) occupy a vital area of this field; porcelain is the material which was made of a mixture of various white – burning clays and fluxes of such nature and in such properties as will produce a dense and vitreous body. Porcelain is classified in two types:-

(1) True or hard porcelain, it is made of kaolin, flint or quartz and fluxes. Hard porcelain has high thermal shock resistance properties, where their properties make it suitable for electrical industry as an insulator.

(2) Soft porcelain; which consists chiefly of a complex glass and may contain very little clay [6].

Electrical porcelains: are chiefly used in insulators and other parts of the electrical applications at ultra high frequencies to specialized problems of direct current; from lowest to highest voltage [2, 6]. In order to establish and maintain high insulating characteristics, porcelain must be thoroughly vitrified and it may be designed for high strength, high voltage application; high frequency [2].

## **1.2 Dielectric Properties**

### **1.2.1 Dielectric Constant**

The capacitance of the parallel plate capacitor, with distance ( $da$ ) and cross – section area  $A$ , in vacuum [7, 8] is given by:

$$C_o = \epsilon_o(A/da) \quad \text{----- (1-1)}$$

Where  $\epsilon_o$  is the vacuum permittivity. When a dielectric material replaces the vacuum between the parallel capacitors, then the capacitance is

$$C = \epsilon(A/da) \quad \text{----- (1-2)}$$

Where  $\epsilon$  is the permittivity of the material. Normally the ability of material to polarize and store electrical charge is described by relative permittivity (dielectric constant),  $\epsilon'$  which may defined as the permittivity of the material to the vacuum permittivity i.e.:

$$\epsilon' = \epsilon/\epsilon_o \quad \text{----- (1-3)}$$

By substitution of equation (1-2) in equation (1-3), the dielectric constant is given in the relation

$$\epsilon' = (1/\epsilon_o)[(da/A)C] \quad \text{----- (1-4)}$$

Where C is the measured value for the charge capacitance of the material. The dielectric constant of ceramic samples is affected by changes in its composition, manufacturing technique and porosity as well as by testing factors including the effect of temperature and voltage frequency.

For many isolated charged particles, the force experienced by a charge located at a position  $i$  relative to other charges, is defined by [9].

$$F_i = q_i \sum \frac{q_j e_{ij}}{r_{ij}^2} \quad \text{----- (1-5)}$$

Where  $F_i$  is the electrostatic force on charge  $q_i$  at distance  $r_i$  from a suitable origin when other charges  $q_j$  are at distance  $r_{ij}$ ,  $\hat{e}_{ij}$  being a unit vector directed from  $i$  to  $j$  [10].

Microscopic field intensity  $\bar{E}_i$  position  $i$  can be then be define by

$$\bar{E}_i = \sum_j \frac{q_j \hat{e}_{ij}}{r_{ij}^2} \quad \text{----- (1-6)}$$

and it is derivable from a microscopic potential  $\Phi_o$  by

$$\bar{E}_i = - \text{grad}_i \Phi_o \quad \text{----- (1-7a)}$$

$$\Phi_o = \sum \frac{q_i}{r_{ij}} \quad \text{----- (1-7b)}$$

The notation  $\text{grad}_i$  indicates differentiation at the point  $i$ . For many charges close together, it is more convenient to deal with charge density  $\rho_c$  instead of the individual point charges  $\rho_c$  is equal to the sum of individual charges in a small element volume  $dV$ . [9] By equality definition

$$\bar{D}_d = \epsilon_o \bar{E} + \bar{P} \quad \text{----- (1-8)}$$

Where  $D_d$  is the dielectric displacement,  $\vec{E}$  is the electric field in the medium, which is defined as  $E = [E_o - (P/\epsilon_o)]$  and P is the polarization [11]. Equation (1–8) describes the macroscopic behavior in terms of three vector quantities [12]; the dielectric displacement D, field E, and polarization P, the dielectric displacement describes the extent to which the dielectric field has been altered by the presence of dielectric. The constant is a measure of the ability of material to store charge relative to vacuum and is a characteristic material property [13, 14, 15].

### 1.2.2 Dielectric loss Index

Firstly the above term is agreed upon internationally, while in the U.S.A., it is formally called the loss factor [14]. The dielectric loss index is defined as the magnitude of imaginary part of relative complex permittivity. The representations of the alternating electric field and dielectric displacement in complex notation [8, 16] is given by:

$$E = E_o \exp(i\omega t) \quad \text{-----(1-9)}$$

$$D_d = D_o \exp.i (\omega t - \delta) \quad \text{----- (1-10)}$$

As the electric field (E) is caused to vary with time, E and  $D_d$  are no longer necessarily in phase, and for sinusoidal fields a complex dielectric constant,  $K^*$ , can be employed in this equation:-

$$D_d = \epsilon E \quad \text{----- (1-11)}$$

Then equation (1–11) become

$$D_d = K^* E \quad \text{----- (1-12)}$$

Where  $K^* = k' - ik'' = \frac{\epsilon^*}{\epsilon_0} = 1 / \epsilon_0(\epsilon' - i\epsilon'')$

$$k^* = k' - ik'' = \frac{\epsilon^*}{\epsilon_0} = 1 / \epsilon_0(\epsilon' - i\epsilon'')$$

Dividing equation (1-10) by (1-9) and then by simplification, one gets

$$\tan \delta = \frac{k''}{k'} = \frac{\epsilon''}{\epsilon'} \quad \text{----- (1-13)}$$

Where  $\tan \delta$  is a loss tangent. This phase shift corresponds to time lag between an applied voltage and induced current, which cause loss current and energy dissipation in circuits which do not require charge carrier migration [16].

The loss index, in terms of electrical conductivity [16] can be written as

$$\sigma = \omega \epsilon'' = \omega \epsilon' \tan \delta \quad \text{----- (1-14)}$$

$$\text{Since } K'' = \frac{\epsilon''}{\epsilon_0}; K' = \frac{\epsilon'}{\epsilon_0} \quad \text{----- (1-15)}$$

$$\text{Its follows that } K'' = \frac{\epsilon''}{\epsilon_0} = \sigma / \omega \epsilon_0 \quad \text{----- (1-16)}$$

By substitution ( $\sigma = 1 / \rho$  ;  $R_p = \rho \cdot (d_s / A)$ ).and by substitution, then

$$K'' = [(1 / R_p \omega \epsilon_0) d_s / A] \quad \text{----- (1-17)}$$

Where  $R_p$  is the measured value for the material resistance [14]. A theoretically perfect insulator would throw current and voltage wave

entirely out of phase, in which case the phase angle  $\varphi_s$  would be  $90^\circ$ , for actual insulator the phase angle is some what less than  $90^\circ$ . Energy losses in dielectrics result an Electronic polarization occurs easily even at frequencies as high as  $10^{16}$ Hz. Since no rearrangement of atoms is necessary. The ion vibration and deformation losses become important in the infrared but are not a major concern for frequencies below about  $10^{10}$  Hz. The far aspect the major factor affecting on the using of ceramic materials is the ion migration losses which tend to increase at low frequencies and as the temperature raised. However, materials that rely no molecular polarization are very sensitive to frequency, since entire atoms or groups of atoms must be rearranged [17].

### **1.3Physical Properties**

#### **1.3.1 Thermal conductivity**

Thermal conduction is the phenomenon by which heat is transported from high–to low temperature regions of substance. In other word, it can be characterized the ability of material for heat transfer from one point to other is the thermal conductivity. It is best defined in terms of expression [18]

$$q = -K \frac{dT}{dx} \quad \text{----- (1-18)}$$

Where q denotes the heat flux or heat flow, per unit time per unit area (area being taken as that perpendicular to the flow direction),  $K$  is the thermal conductivity coefficient,  $\frac{dT}{dx}$  is the temperature gradient through the conductivity medium [18]. Thermal conductivity is a property which is, as yet not fully explained on the basis of theoretical reasons. Debye considers that the atomic or ionic groups with in a crystal act as oscillators

or vibrators and if these frequencies correspond to those of thermal waves, conduction will be proceed. On this basis, highly symmetrical crystals where the vibrations are harmonic will be better conductors of heat than those which are disordered or contain many types of bond. Whilst this simple concept applies in general terms, there are many discrepancies in experimental finding which suggest that other factors are involved.

The main factors to be considered in connection with the rate of passage of heat transfer through ceramic materials are: - the chemical composition of the material used, its previous heat treatment, the texture or physical condition of the material, the porosity of the material and the sizes and distribution of the pores, and the temperature at which the material is used or tested. The influence of texture and porosity may be considered together, as the principle effect on the thermal conductivity is due to the relation between the amount of solid and air which the heat has traverse in passing through the material. As air is a much better insulator than any solid material, the larger the proportion of the air the greater will be thermal insulating power of the material. Hence, a fine-grained, close – textured material has a much greater thermal conductivity than one with a coarser open texture. The relation between insulating power and texture or porosity cannot, however, be expressed in very simple terms, as it is modified by (a) temperature,(b)the size, (c)the shape of the pores or interstices, and (d) the position of the interstices relative to each other and the solid matter[6].

### 1.3.2 Measurements of Thermal Conductivity

The less disc is a useful method for measurements of thermal conductivity for the prepared samples of our study [19]. Where the unknown sample is usually sandwiched between two reference discs of known conductivity and the temperature differences across each of disks are determined. The less disc schematic diagram is represented in fig. (1-1) was used in measuring the thermal conductivity [20].

This method was used for measuring the thermal conductivity of the samples produced in this study. Sample of 27mm diameter was inserted the two discs A and B with equal diameter( $d_A$ ). The heat supplied from heating coil built in disc inserted between C and B. The supplied current (I) was constant for all measurements and its value was 0.25A, and voltage was 6 volts. The temperature of disc A, B, which are represented by  $T_A$  and  $T_B$  measured in °C. Then the thermal conductivity of testing sample is calculated by this equation [11]:

$$K=e [T_A + (2/r_d) (d_A + d_s/2) T_A + d_s T_B / r_d] / (T_B - T_A / d_s) \quad \text{----- (1-20)}$$

Where  $r_d$  is the sample radius (mm)

$d_s$  is the sample thickness (mm)

$e$  is the emitted energy ( $w/m^2 \cdot ^\circ C$ )

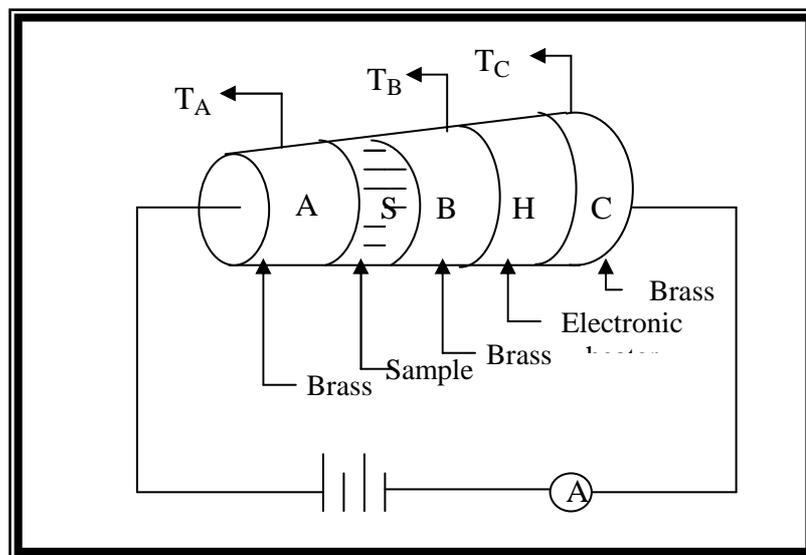


Figure (1 - 1) Less disc (Ref.20)

## **1.4 Mechanical Properties:**

### **Compressive Strength**

The mechanical behavior of materials described by their mechanical properties; these tests are designed to represent different type of loading condition [1]. Ceramics have comparable tensile and compressive strength [21], in this work we will study compressive strength property. If a force, F, applied to the cross section area of a bar A, we obtain normalized term called the stress, thus [22]

$$\sigma = \frac{F}{A_0} \quad \text{----- (1-21)}$$

Where  $\sigma$  is Compression strength in (MPa).

$A_0$  is the original cross-section of the sample in (m).

F is the normal force in (N).

## **1.5 Infrared Analysis:**

Infrared spectroscopy can be described as the use of instrumentation in measuring a physical property of matter, and then relating the data to chemical composition. The instruments used are called infrared spectrophotometers and physical property measured is the ability of matter to absorb, transmitted, or reflect infrared radiation [23]. The basic principle of infrared instruments is to measure the vibrational spectrum of a sample by passing infrared radiation through it, that wavelengths have been absorbed and what extent. Since the amount of energy absorbed is a function of the number of molecules present. The source of infrared may be a glowing filament or hot silicon carbide rod, both of which emit radiation over a wide range of frequency. The infrared beam passes through a collimator to the specimen which should be in the form of a thin film. The transmitted rays then pass through a system of mirrors on the

rock salt prism, which can be rotated, so vary the frequency of radiation received on the detector [6].

The analysis for these spectrums was carried out using two frequency regions. The details are as following:

### **1 –Wave number between 3500 and 3700 cm<sup>-1</sup>:-**

Ruswell and Dudenbostel [24] first showed that absorption in this range is due to hydroxyl group (O–H). In the structure of dioctahedral minerals, each pair of aluminum ions shares two hydroxyl groups, which are related by a center of symmetry between the aluminum ions. Sarasota [25] through that the absorption frequency of (O–H) bonds depends on the degree of association of these groups. Miller [26] concluded that the bonds at 3718 and 3677 cm<sup>-1</sup> for kaolin are due to stretching vibrations of relatively unassociated (O–H) groups, i.e due to hydraxyle groups nearly free of hydrogen bounding to the other atoms.

In general, the layer–silicate structural OH groups that are comparatively slightly show absorption of high frequencies of 3600 to 3700 cm<sup>-1</sup>, where as the absorbed water shows absorption at low frequencies 3400 cm<sup>-1</sup>, and another band 1640 cm<sup>-1</sup> corresponding to the deformation vibration of water[27].

### **2 Wave number between 1500 and 600 cm<sup>-1</sup>.**

The position and sharpness of the perpendicular vibration varied with physical state; thus in the spectra of kaolin of large crystal size, the node appears as a broad shoulder near (1080) cm<sup>-1</sup> [27, 28]. The band intensities and shift to higher frequencies in the spectra of smaller crystals until in very finely ground material the band is at 1109 cm<sup>-1</sup> smaller but distinct shifts in frequency, together with sharpening and intensification of bands as particle size decreases [11].

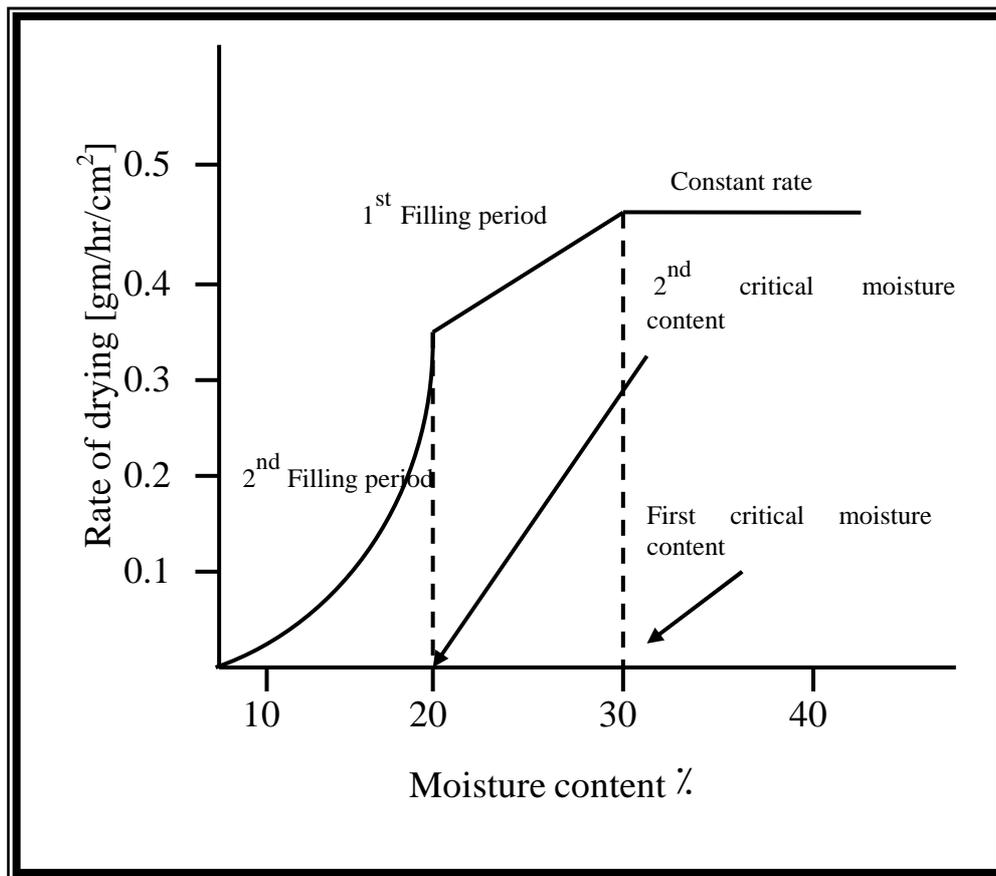
## **1.6 Process Parameters**

### **1.6.1 Mixing**

The mechanism of mixing is influenced by many factors such as the shape and size of the mixing vessel, the physical properties of the solid particle and the operating condition [29,30]. Mixing, an important operation in chemical process industries can be divided into five areas: – liquid–solid dispersion, gas–liquid dispersion, liquid–liquid dispersion, the blending of miscible liquids, and the production of fluid motion. Mixing performance is evaluated by two criteria, the first is physical uniformity, i.e., a physical relationship required in terms of samples of uniformity in various parts of mixing vessel, the other criterion is based on mass transfer or chemical reaction. The elements of mixer design are the process design fluid mechanics of impellers, impeller power characteristics–relate impeller power, mechanical design shafts and drive assembly [29].

### **1.6.2 Drying**

Drying can be defined as the removal of water from a granular material by evaporation [31]. This process involves both the transfer of heat from the surrounding environment to the solid–water system and the simultaneous transfer of water vapor in the reverse direction [32]. Drying is accomplices by shrinkage, which tends to bring the particles so close together that attractive forces become so strong that water can no longer penetrate between them, the wetting and moderate drying, frequently tend to increase the plastic properties of clay mineral [33]. Fig. (1–2) represents the drying behavior expressed in terms of the drying rate versus the moisture content percentage [34].



*Figure (1–2). Rate of drying curve [34].*

On such a curve three periods can be distinguished, the first: – The constant rate period, during which the rate of evaporation is independent of moisture content. The second is the first filling–rate period, during which the rate of drying is very often a linear function of the moisture content, and the third is the second filling–rate period, with a curvilinear relation between rate of drying and moisture content. The constant–rate period and the first falling–rate period ends are termed the first and second moisture content respectively[34].

### 1.6.3 Shrinkage

Shrinkage on drying is a profound concern to the structure clay products industry. Since clay minerals are responsible for shrinkage, the amounts percentage and their particles sizes determine the shrinkage potential; then, the amount of water present in the plastic clay is proportional to, but not equal to shrinkage. Differential shrinkages set up stresses during drying that can cause warping and cracking of the sample. Variations in moisture content in the piece are largely responsible for shrinkage differences from one part of the body to another. Another unique feature of clays is increased strength on drying, the shear strength of clay bodies increases from the plastic strength to a maximum when all the absorbed water is removed.

During drying, large dimensional changes occur as fig. (1-3) Most of the shrinkage occurs during the initial stages of drying as the inter particle water evaporates; the temperature and humidity are carefully controlled during drying to minimize stress, distortion, or cracking [1].

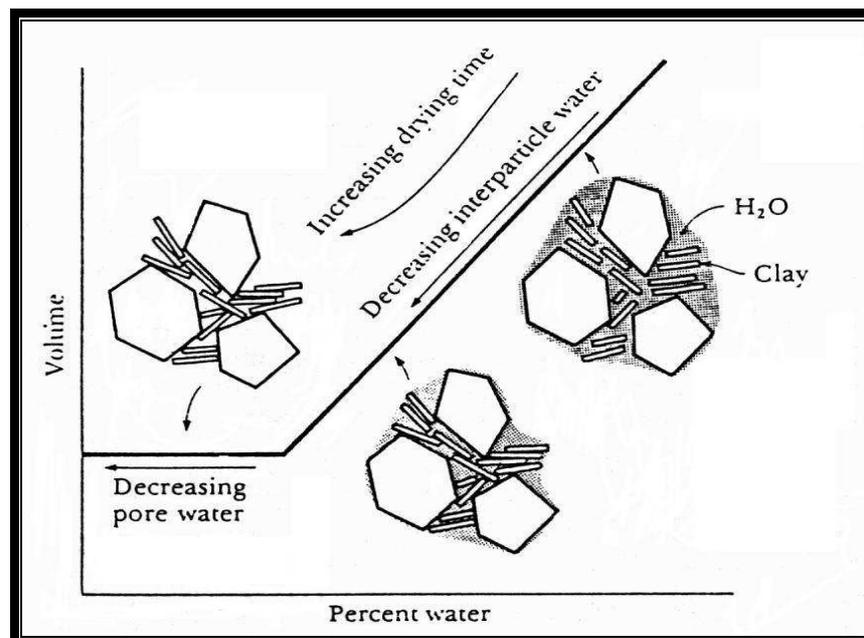


Figure (1-3). The change in the volume of ceramic body as the moisture is removed during drying [1].

Other factors also influence shrinkage, one of these is body thickness; non uniform shrinkage and defect formation are more pronounced in thick pieces than in thin ones. Water content of the formed body is also critical; the greater the water content, the more extensive the shrinkage. Consequently, the water content is ordinarily kept as low as possible. Clay particle size also has an influence; shrinkage is enhanced as the particle size decrease, to minimize shrinkage, the size of the particle may be increased, or nonplastic material having relatively large particles may be added to the clay [18]. The value of linear firing shrinkage is, in the percent of shrinkage, due to variations in the size and shape of sample particles, linear shrinkage is approximately proportional to the inverse of the particle radius but is not greatly affected by sintering time [35].

Size distribution is important in practical system where shrinkage is to be minimized. Mostly large particles and sufficiently small particles to fill in the interstices will give a particle compact of highest green density. The linear firing shrinkage also varies with composition and tends to increase as fluxing components increase with the production of relatively more glassy material and less-high-temperature crystalline phases during the firing [34,36].

#### **1.6.4 Sintering &Firing**

Sintering is common term for the process by which a slight ( non-densification ) or large (densification ) reduction in pore volume occurs when heating a powder compact to temperatures close to melting [37].The process has been recognized as a very complicated evolution of microstructure through the action of several different transport mechanisms. The most general concept of sintering is the interface elimination process, i.e. the particles sinter together by atomic motions that act to eliminate the high surface energy associated with an unsintered

powder, therefore, during the process the total free energy of the system would decrease.

As important step in production of most ceramic products in firing, where the purpose is to agglomerate particles into mass by sintering, the sintering operation brings about several significant changes in ceramic product: —

- (1) the total surface area is reduced.
- (2) the bulk volume is reduced
- (3) the strength is increased.

The dimensional changes which occur during firing are just as important as those which occur during drying; in fact, probably more so, because shrinkage take place in almost all aggregated materials during firing [21]. During firing, the rigidity and strength of ceramic increase firing or sintering, cause additional shrinkage of the ceramic body as the pore size between the particles is reduced. We must control four features of the final sintered microstructure—grain size, pore size, pore shape, and amount of a glass.

During sintering, ion first diffuses along grain boundaries and surfaces to the points of contact between particles, providing bridging and concenection of the individual grains. Further grain boundary diffusion closes the pores and increases the density, while the pores become more rounded. Firing initial particle size and higher temperatures accelerated the rate of pore shrinkage, when the pores become so small that they no longer pin the grain boundary, grain growth occurs as fig. (1–4) [1].

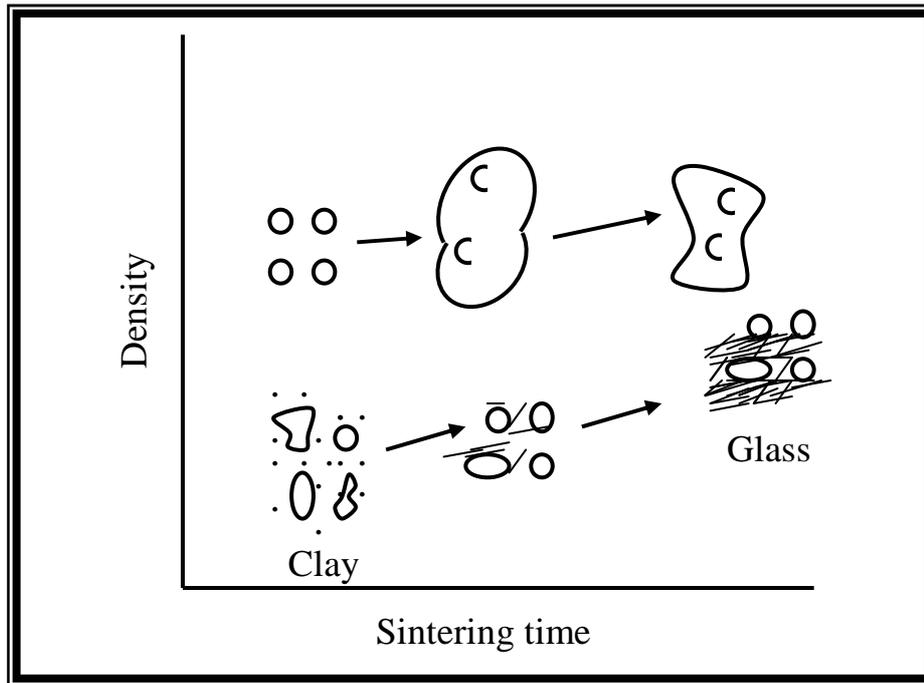


Figure (1–4). During sintering, diffusion produces bridges between the particles and event causes the pores to be filling in [1].

## 1.7 Raw Materials

### 1.7.1 Kaolin

The term kaolin is coming from the Chinese word kaoliang (meaning “high ridge “), from which the clay originally came [38]. The kaolin’s groups are koalinite, nacrite, dickite and halloysite. Their basic structure consist of oxygen atoms arranged to give alternate layers of tetrahedral holes and octahedral holes. Where these layers are filled with silicon in tetrahedral holes and aluminum in two–third of the octahedral ones we get the common mineral kaolinite, and more perfect and rarer minerals dickite and nacrite. The structure applies equally to all the members of kaolin group; the distribution feature in the way in which the units are stacked one upon another[11]. This would give theoretical composition of 39.8 %Al<sub>2</sub>O<sub>3</sub>, 46.3 %SiO<sub>2</sub>, and 13.9% H<sub>2</sub>O. [38].

### 1.7.2 Silica

The silicate structures are based on the silica tetrahedron. Silica tetrahedral,  $\text{SiO}_4^{4-}$ , Behave as ionic group; the oxygen ions at the corners of the tetrahedral are attached to other ions or ionic groups to satisfy the charge balance [1]. When all four corners of the tetrahedral are shared, silica, or  $\text{SiO}_2$  is produced, as the temperature increases, silica changes from  $\alpha$ -quartz to  $\beta$ - quartz to  $\beta$ -tridymite to  $\beta$ -cristobalite to liquid. The pressure-temperature equilibrium diagram in fig. (1-6) shows the stable forms of silica.

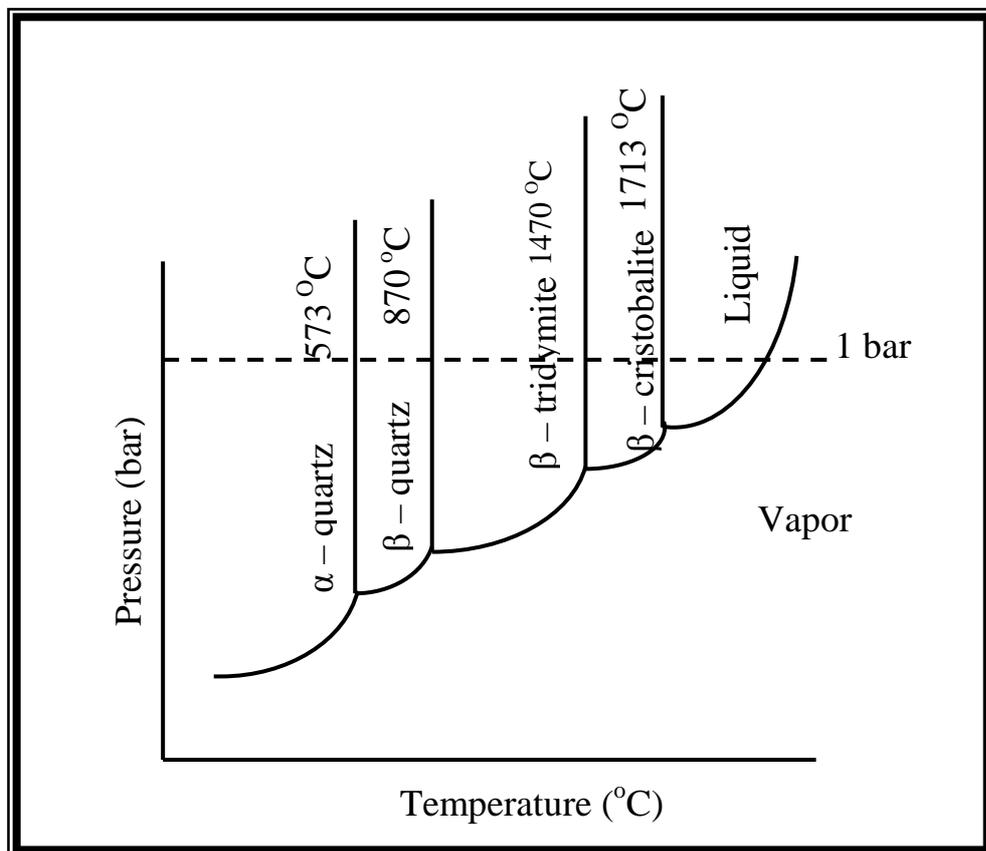


Figure (1-5). The pressure – temperature phase diagram for  $\text{SiO}_2$  [1].

The transformation from  $\alpha$  – quartz to  $\beta$  – quartz is a displacive transformation. The quartz rapidly changes crystal structure by slight distortion of the lattice involving second or further nearest neighbors. Similar transformations occur between different forms of tridymite and cristobalite. The transition between  $\beta$  – quartz and  $\beta$  – tridymite and between  $\beta$  – tridymite and  $\beta$ –cristobalite are reconstructive rather than displacive. A reconstructive transformation requires that bonds between the atoms be broken and reestablished to produce a major change in crystal structure [1].

### 1.7.3 Feldspar

The most common constituents of igneous rock formations are the feldspars. These important minerals are used extensively in the manufacture of porcelains [2]. Feldspars are used in the fine ceramic industry as a flux to form a glassy phase in bodies [6].

There are two clearly – defined groups:-

- (1)Based on orthoclase  $\text{KAlSi}_3\text{O}_8$ , orthoclase was used in general as K – feldspar with monoclinic optical properties [2 –39].
- (2)Termed the plagioclase group, which are solid solution of albite,  $\text{NaAlSi}_3\text{O}_8$ , and anorthite,  $\text{CaAl}_2\text{Si}_2\text{O}_7$ ; celsian  $\text{BaAl}_2\text{Si}_2\text{O}_8$ , which is not of common occurrence, is similar to orthoclase.

The structure of all feldspars is based on framework of oxygen–silicon tetrahedrons sharing four corners, there is replacement of silicon by aluminum ions, however which results in charge–deficiencies, balanced by cations of  $\text{K}^+$ ,  $\text{Na}^+$ , or  $\text{Ca}^{2+}$  entering the lattice. The relative ionic radii of these cations are  $1.33 \text{ \AA}$ ,  $0.98 \text{ \AA}$ , and  $1.06 \text{ \AA}$  respectively, the structure varies with the cation. The feldspar minerals with small  $\text{Na}^+$  and  $\text{Ca}^{2+}$  are considered as a “collapsed “ structure and thus lose some symmetry to

make them triclinic, on the other hand, the  $K^+$  minerals are generally monoclinic [2– 6].

#### **1.7.4 Zinc Oxide "ZnO"**

Zinc is valuable auxiliary flux in glazes firing up to about 1050 °C. At higher temperatures it is too active in reducing the viscosity. It reduces the coefficient of thermal expansions and improves the chemical durability of some compositions. Zinc oxide is usually produced by direct oxidation of zinc metal or high zinc alloy, calcined zinc oxide is often used where there is a need to minimize shrinkage during drying and firing [40]. So may additive more used a mineralized, only ZnO causes a considerable action on the mullite content [4].

#### **1.7.5 Binder**

The selection and application of organic or inorganic binder can be classified as the second most critical factor in the dry pressing process. The term “binders” loosely covers all pressing aids such as binder’s lubricants, plasticizers and deflocculates [41]. The binders are dissolved molecularly in water or an organic solvent or are dispersed in liquid as an “emulsion “. The liquid phase is important for uniformly dispersing the binder throughout the particulate.

Polyvinyl Al–cohol (PVA) binder has stable viscosity to improve the forming behavior of powder. The behavior of agglomerates depends on the type, quantity, and properties of the binders, which produce soft agglomerate, PVA does not ionize, but with increasing concentration and raising viscosity. Low molecular weight PVA dissolves more readily, but considerable stirring is necessary with higher molecular weight polymers to complete the dissolution [42].

### **1.7.6 Sodium Carbonate " $\text{Na}_2\text{CO}_3$ "**

Sodium Carbonate is a white, crystalline and hygroscopic powder with a purity of  $> 98\%$ . Sodium Carbonate has melting point of  $851\text{ }^\circ\text{C}$ , it decomposes when heated at temperature greater than  $400\text{ }^\circ\text{C}$ , and sodium carbonate is a strong alkaline compound with pH of 11.6 for 0.1 M aqueous solution [43]. Tensile strength and elasticity are decreased as the content of soda increase of sodium although to a lesser degree than with the other alkalis. Small increases of sodium oxide in a glaze will increase the coefficient of thermal expansion and reduce the softening point with rapid and noticeable consequences to the glaze body fit [40].

### **1.6.7 Sodium Silicate " $\text{Na}_2\text{SiO}_3$ "**

This is a widely used reagent, particularly in conjunction with sodium carbonate. Sodium silicate is inorganic hardeners, have an efficient binding action but have significant side effects on other rheological properties (such as deflocculation) [40].

### **1.8 Aim of Thesis:-**

One of additives, which are used in the produce of porcelain bodies is the electrolyte solution. Most of the previous studies in this field did not give the exact values of these additives moreover it was not examined with local materials. Therefore this study was carried out to optimize the required weight percentage (wt %) of these additives to be applicable to porcelain body, which is prepared from Iraqi clays, to be used an electrical insulators. This can be done by investigating the effect of using electrolyte solution with following concentration (1%,07%,0.5%, 0.2%,0.1%) with fixed percentage of mineralizer material (2%ZnO) , and by studying the electrical properties(dielectric constant & dielectric loss index),for two

sintering temperature (1300°C, 1350 °C) and study Physical properties (Thermal conductivity),and Mechanical properties (Compression strength) using three sintering temperatures(1250 °C,1300°C, 1350 °C), and select the best concentration of electrolyte solution using in electrical insulators industry.

## *Chapter Two*

### *Electrolyte Solution*

#### **2.1 Introduction**

Electrolytes are class of solid solutions that exhibit special behavior compared to non–electrolytes. The distinction arises both because electrolytes dissociate upon dissolution and because the ions produced interact through much large distances than uncharged solutions[44]. It is well known that many substances– inorganic salts in particular–dissociate to form ions in aqueous solutions. The most direct evidence of this is the large electrical conductivity of such solution; in fact, the solution are called electrolytes because they conduct electricity readily [45].

When finely ground material is dispersed in water there is an adsorption of ions on the surface of the constituent particles, one type of ion (either positive or negative) attaching itself to the solid and the other, counter ion, free in water yet held in close proximity by attraction. As a consequence the solid particles become charged [46].

#### **2.2 Clay–Electrolyte System:**

Clay minerals are the main component of suspended solid material in natural waters, responsible for the transport of inorganic and organic contaminants. Electrolytes, dissolved and particulate organic matter, and inorganic particulates, comprised of clays, quartz, carbonates, and a variety of metal oxides (most frequently Al, Fe and Mn) [47].

A colloidal solution may be defined in general terms as a suspension of particles in liquid, where the grain size of the solid is so small that they will not settle; yet at the same time they cannot be regarded as being in true solution. Various forces act on colloidal particles suspended in a medium, and these determine the behavior of slips and slurries which are essentially

fine-particles suspensions, like Van der Waal forces, Gravitational forces, thermal movement and Colloidal particles. Colloidal particles when suspended in water are charged and are surrounded by a water shell. The molecules of water within this shell are rigidly held by particle and have a different character and properties from those in the remainder of the dispersion medium [6].

The DLVO (Derjaguin–Landau–Verwey–Overbeek) theory [48] explains the stability of colloids by looking at two opposing forces acting upon colloidal particles. These two forces are the electrostatic repulsion explained by the double layer model and the Van der Waals forces which are weak attractive forces between particles. This theory explains the tendency of colloids to agglomerate or to remain in a stable state of dispersion by combining the two forces. The net interaction effect, shown in fig. (2–1) is the result of the subtraction of the attraction force from the repulsion force. If the net value is positive a repulsion effect will be observed. In this figure it can be observed how, by increasing the natural Zeta Potential (ZP), the natural ZP typically found in colloids in aqueous suspension ranges from  $-14$  mV to  $-30$  mV. At negative charge values higher than  $-30$ mV enough repulsion occurs to favor a stable dispersion. The more negative the charge value, the stronger the dispersion effect. For values ranging between  $-45$ mV and  $-70$ mV stable dispersions are assured. As the ZP approaches zero, the repulsion effect is lost and agglomeration begins. For values ranging from  $-10$  mV to  $-15$  mV a threshold of agglomeration is observed, and from values between  $-5$  mV to  $+5$  mV strong agglomeration occurs [48].

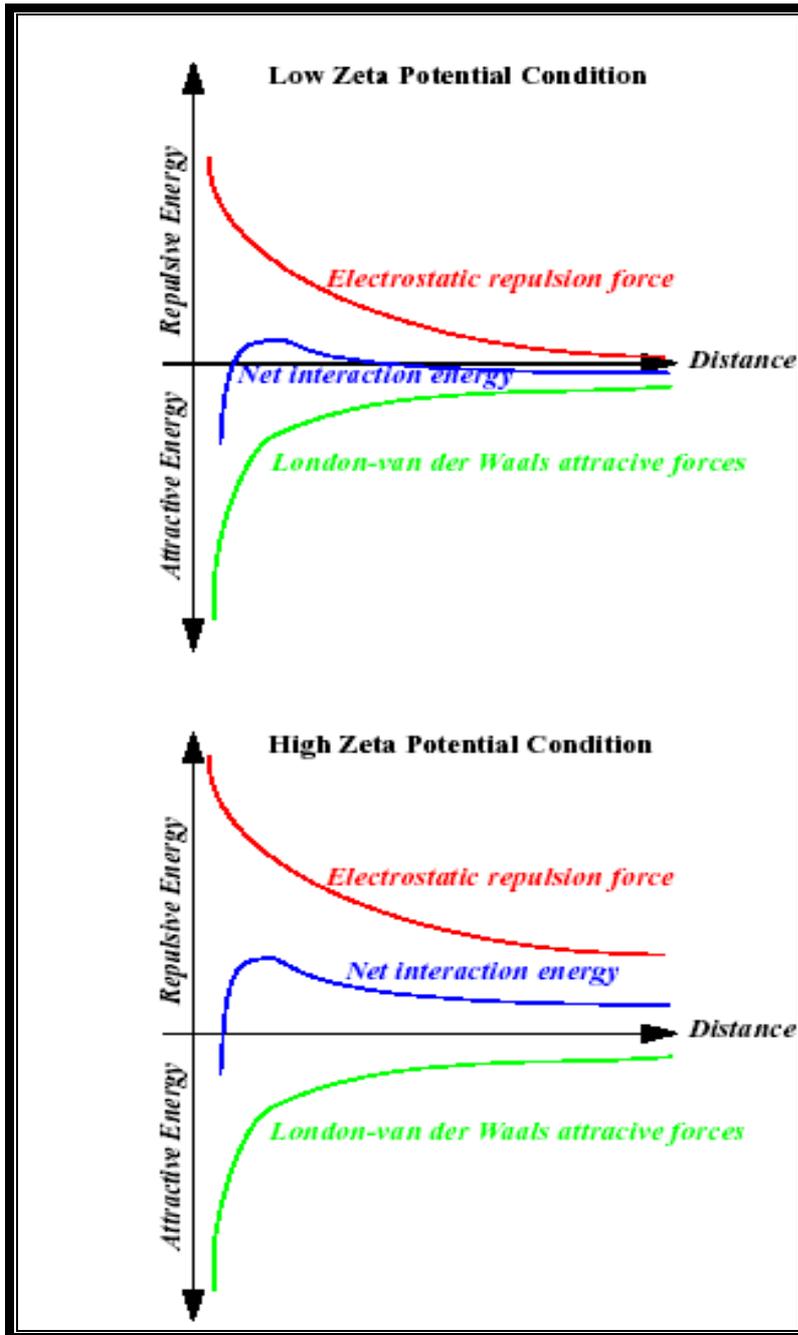


Figure (2 –1) Net Interaction Energies at low and high Zeta Potential [48].

When a sol is colloiddally unstable, the formation of aggregates is called coagulation or flocculation. These terms are often used interchangeably, but some authors prefer to introduce a distinction between coagulation, implying the formation of compact aggregates, leading to the macroscopic separation of a coagulum; and flocculation, implying the

formation of a loose or open network which may or may not separate macroscopically. In many contexts the loose structure formed in this way is called a floc. The reversal of coagulation or flocculation, i.e. the dispersion of aggregates to form a colloidal stable suspension or emulsion, is called deflocculation. The rate of aggregation is in general determined by the frequency of collisions and the probability of cohesion during collision [49]. The deflocculating electrolyte increases the electrostatic forces of repulsion on receptive particles so that they move apart flocculating salts have the opposite effect. Fine particles no longer repel each other but aggregate into flocs [40].

The salt concentration is so high that aggregates no longer are Brownian, and sediment, giving a clay–water "phase separation". The sedimentation is the settling of suspended particles under the action of gravity. If the concentration of particles is high and interparticle forces are strong enough, the process of sedimentation may be better described as compaction of the particle structure with pressing out of the liquid. This particular kind of settling is also called subsidence. Sediment is the highly concentrated suspension which may be formed by the sedimentation of a dilute suspension [49]. Sedimentation level at given clay concentration decreases as the electrolyte concentration is increased, as the DLVO theory suggests [50].

### **2.3 Electrical double layer:**

When a charged colloidal particle is immersed in an electrolyte solution, it is surrounded by counterions—i.e.; small ions of opposite sign—to balance the surface charge. The charged colloidal surface along with the neutralizing diffuse layer of counterions is often referred to as the electric double layer EDL, which plays an important role in many aspects of interfacial phenomena [51].

The double layer theory model explains the repulsive forces between colloids. It is focused on the effect that the negatively charged colloids have over the positive ions in the bulk solution. The positive ions (counterions) form a firmly attached layer around the surface of the colloid known as the Stern Layer ( $\delta$ ). Additional positive ions attracted by the negatively charged colloidal particle face a repulsion force from the counter-ions attached on the Stern layer as well as by other counter-ions approaching the colloid. The density of this layer, known as the diffuse layer, gradually decreases with distance from the colloidal particle, until it reaches equilibrium with the rest of the ions in the solution. It is the diffuse layer surrounding the colloid that creates the most far reaching repulsive force between colloidal particles; the higher the density of the diffuse layer, the greater the distance over which these repulsion forces are significant[52] as fig .(2–2),  $K$  is the thickness of electrical double layer is known Debye length, this Debye length is also called a screening length as it gives an estimate at what distance away from the surface the surfaces charges are screened by the diffuse counterion layer. The magnitude of the Debye length depends exclusively on the properties of the liquid and not on any property of the surface [53]. The electrical double layer is formed at interfaces of charged objects and electrolyte solutions composed of ions and solvent molecules. The ions with the charge of the opposite sign than the charged object (counterions) are accumulated close to the charged

object, while the ions with the charge of the same sign as the charged object, (cations) are depleted from the vicinity of the charged [53].

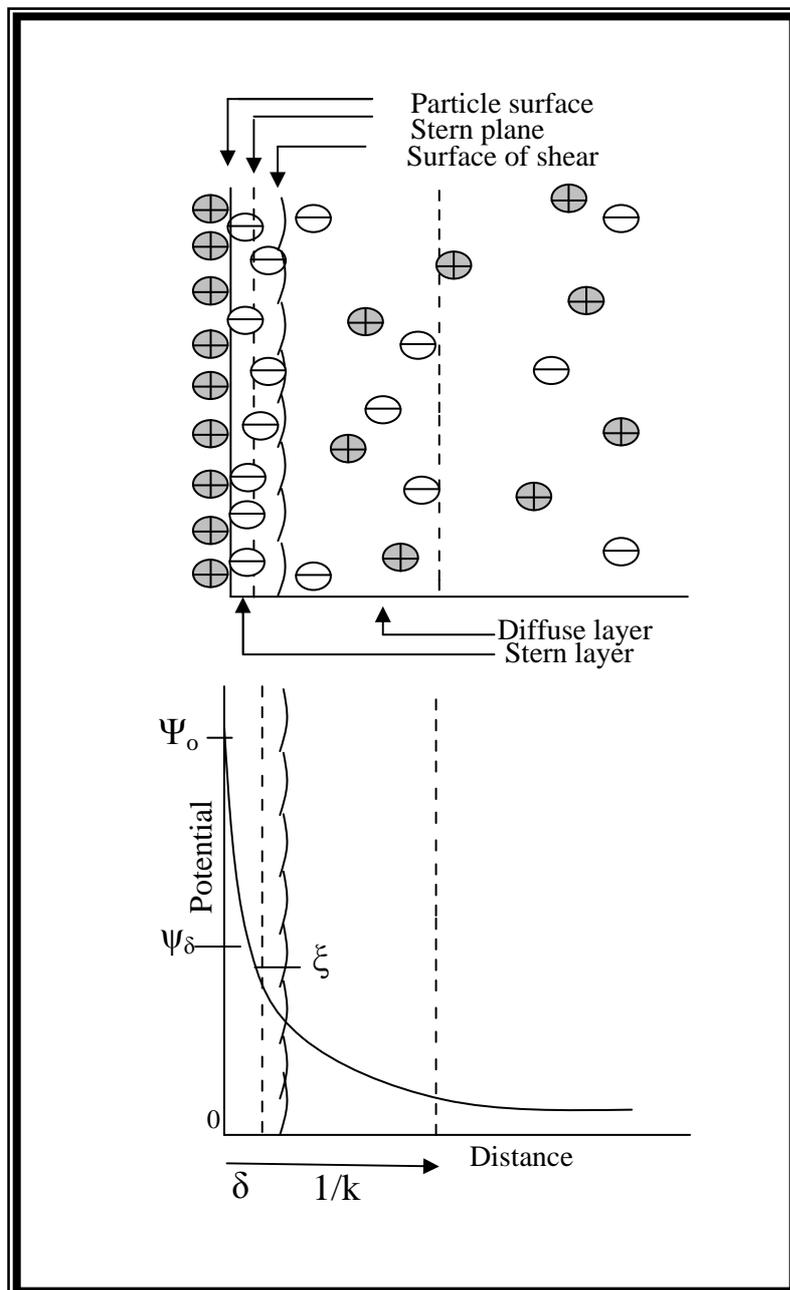


Figure (2-2). Schematic presentation of the distribution of dissociated charges expanding into the liquid phase [51].

## **2.4 Swelling:**

The swelling of clays can be discussed in terms of particle interaction. Two stages of swelling should be distinguished. The first stage, up to four monolayer of water penetrate between layers of expanding clay, or between flat surfaces of adjoining particles. In the parting of the surfaces, three forces are operative—the van der Waals attraction, the electrostatic interaction of charged surfaces and cations, and the adsorption energy of the water.

From the net interaction energies, the forces operating in the stage of swelling are derived to be of the order of a few thousand atmospheres for the entrance of the first monolayer of water. The forces decline rapidly with the entrance successive water layers. In the second stage of the swelling process in which the plates are separated to distances larger than equivalent with the thickness of four layers of waters, the swelling is now governed by double layer repulsion, which is identical with the “osmotic pressure” of the system[54]. Seen that these micropores vary in shape and size with changes in chemical potential and electrolyte type. The electrolyte type affects both, the number of platelets in each stack and the average size of micropores as fig. (2 – 3) the treatment of electrolyte type effects on the basic size of micro pores, with the decrease in the bulk liquid potential (more negative), both platelet spacing and micropore size decrease [55].

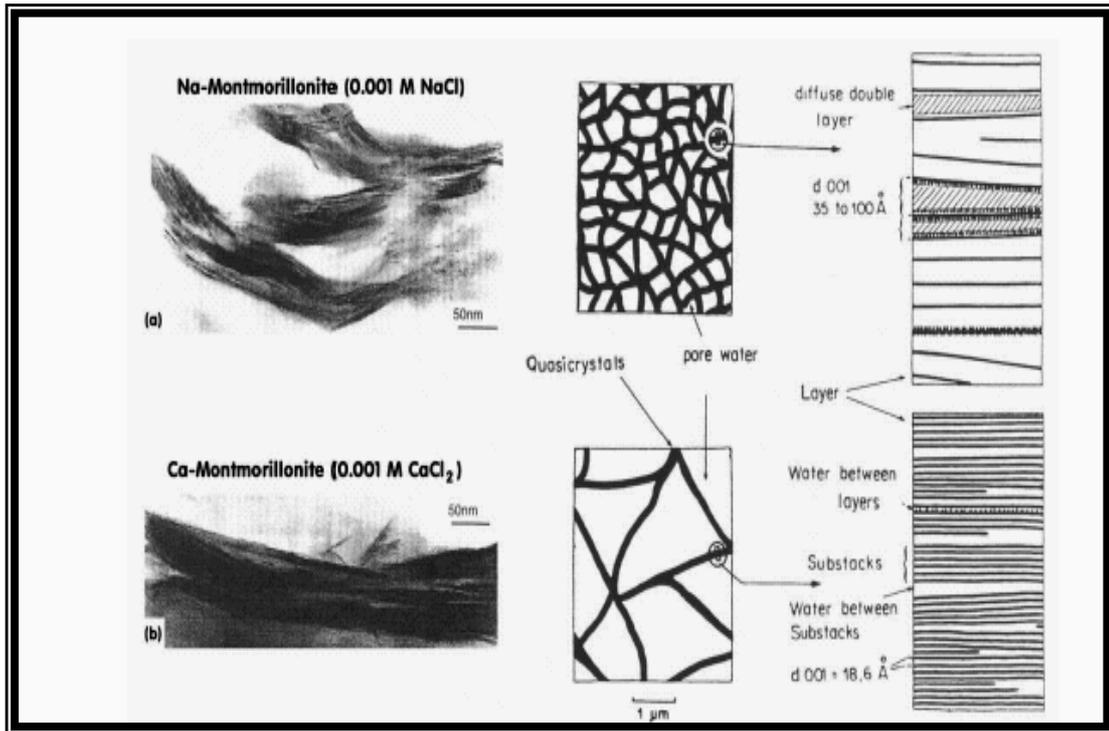


Figure (2–3) Clay fabric microstructure as affected by electrolyte solution composition [55].

## 2.5 Ion Exchange

Clay minerals have the property of sorting certain anions and cations, retaining these in an exchangeable state; i.e. they are exchangeable for other anions or cations by treatment with such ions in a water solution (the exchange reaction also take place sometimes in anon aqueous environment). The exchangeable ions are held around the outside of silica–alumina clay mineral structural units, and the exchange reaction generally does not affect the structure of silica–alumina clay packet. In clay minerals the common exchangeable cations are calcium, magnesium, hydrogen, potassium, and sodium, frequently in about that order of general relative abundance [33].

## **2.6 Ionic Strength**

Electrolytes containing ions with multiple charges have larger effects on the active coefficients of ions than electrolytes containing only singly charge ions, ionic strength is the quantity representing interactions of ions with water molecules and other ions in solution. In order to express electrolyte concentration in a way that takes in account introduced the ionic strength [47] I defined by

$$I = \frac{1}{2} \sum_i m_i z_i^2 = \frac{1}{2} (m_1 z_1^2 + m_2 z_2^2) \quad \text{----- (2-1)}$$

Where I is the ionic strength.

m is molar concentration.

Z is valance number.

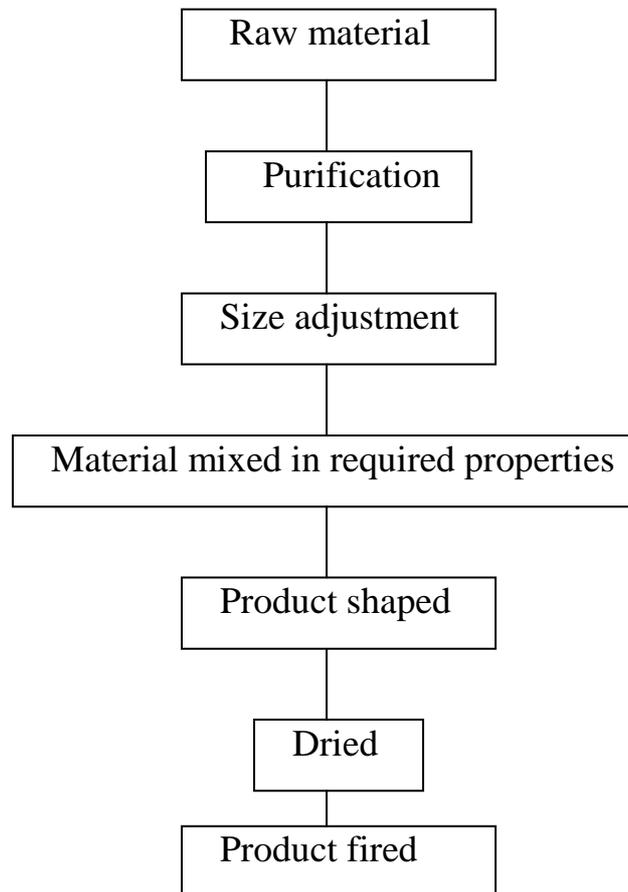
Solution ionic strength influences the dynamics of colloidal deposition and transport in heterogeneous porous media by controlling the range and the magnitude of interparticle forces. Low to moderate concentrations of indifferent electrolytes containing monovalent counterions promote interparticle repulsion and declining deposition rates as accumulated particles block collector surfaces from subsequent deposition. The presence of specifically interacting, divalent counterions at moderate concentrations promote interparticle attraction, and the presence of rising deposition rates as particles retained on stationary mineral grains act as additional collectors. A reduction in ionic strength produces an expansion of the electrical layer surrounding charged colloidal particles, there by causing repulsion between particles deposited on favorable patches and reduction in the maximum attainable surface coverage corresponding to a monolayer of deposited particles [56].

## *Chapter Three*

### *Practical Part*

#### **3.1 Introduction**

Ceramic may be defined as a product manufactured by the heat treatment of a material or a mixture of materials, which are inorganic and non-metallic. A generalized diagram of major process involved although in particular cases some of these stages may be unnecessary or other required may be following[57, 58]



*Figure (3– 1) Flowchart of process diagram*

### 3.2 Experimental Work:

*1-Raw Material Treatment &Characterizations:* - The raw materials are used as a matrix composition; Table (3-1) shows the chemical composition of these materials. The phase structure for these materials are measured by XRD Tech. by using (SIEMENS X-RAY DIFFRACTION, UNIT MODEL D – 500, KV = 40, CU K $\alpha$  = line,  $\lambda$  = 1.542 $^{\circ}$ A). These results are shown in fig. (3-2), (3-3) and (3-4) respectively for these materials.

*Table (3-1) Chemical composition for raw materials*

| Material                       | Kaolin Duekhla | Ardhuma sand | potash feldspar |
|--------------------------------|----------------|--------------|-----------------|
| oxide                          | percentage     | percentage   | percentage      |
| SiO <sub>2</sub>               | 52.35          | 97.39        | 69.61           |
| Fe <sub>2</sub> O <sub>3</sub> | 1.31           | 0.08         | 0.19            |
| Al <sub>2</sub> O <sub>3</sub> | 34.02          | 0.38         | 15.07           |
| TiO <sub>2</sub>               | 0.12           | —            | —               |
| CaO                            | 1.2            | 0.246        | 0.98            |
| MgO                            | 1.11           | 0.015        | 0.13            |
| SO <sub>3</sub>                | 0.45           | —            | —               |
| Na <sub>2</sub> O              | —              | 0.18         | 3.25            |
| K <sub>2</sub> O               | —              | 0.03         | 8.35            |
| L.O.1                          | 12.54          | 0.25         | 0.51            |

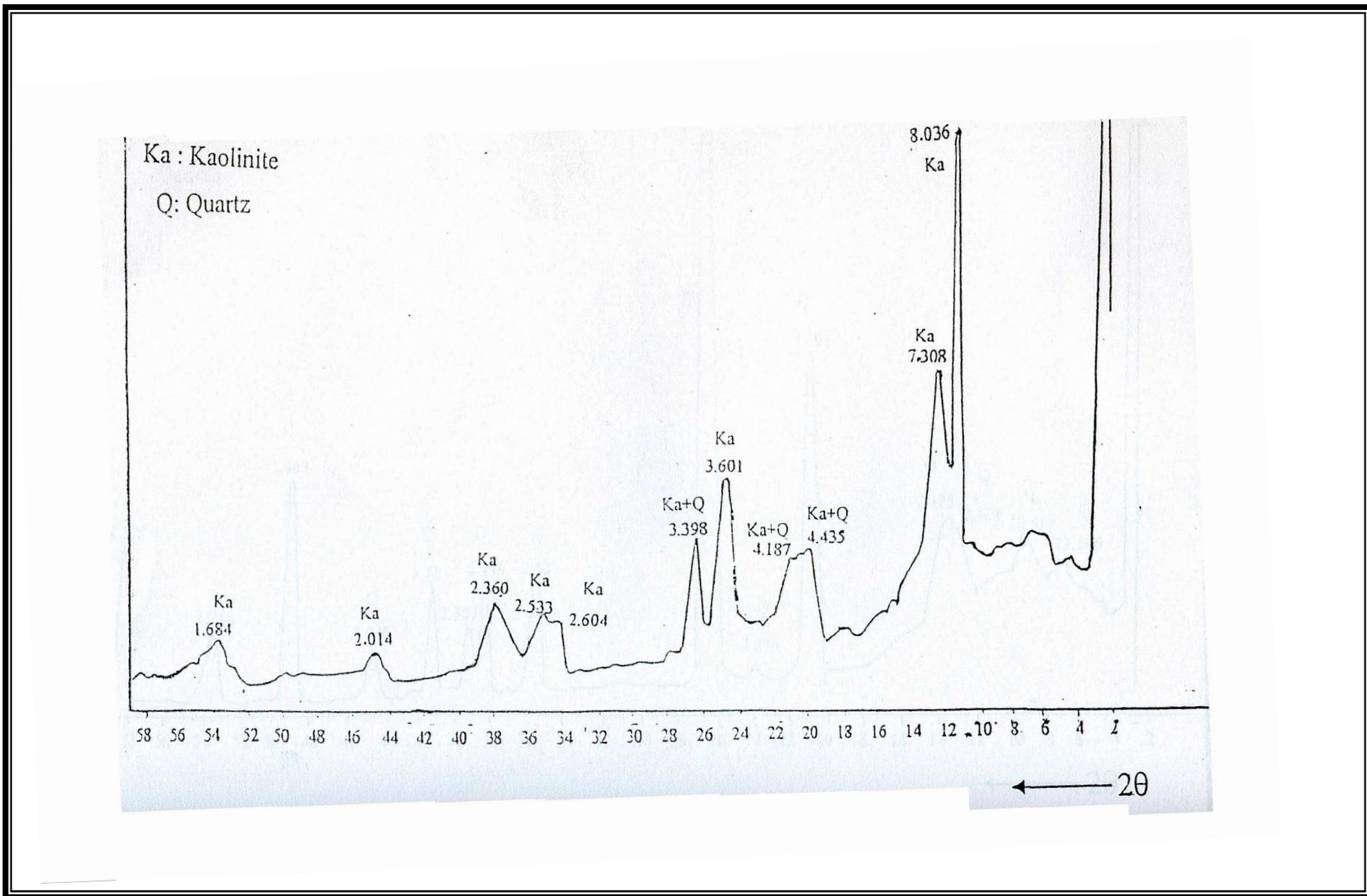


Figure (3-2) XRD pattern for Kaolin Duekhla raw material

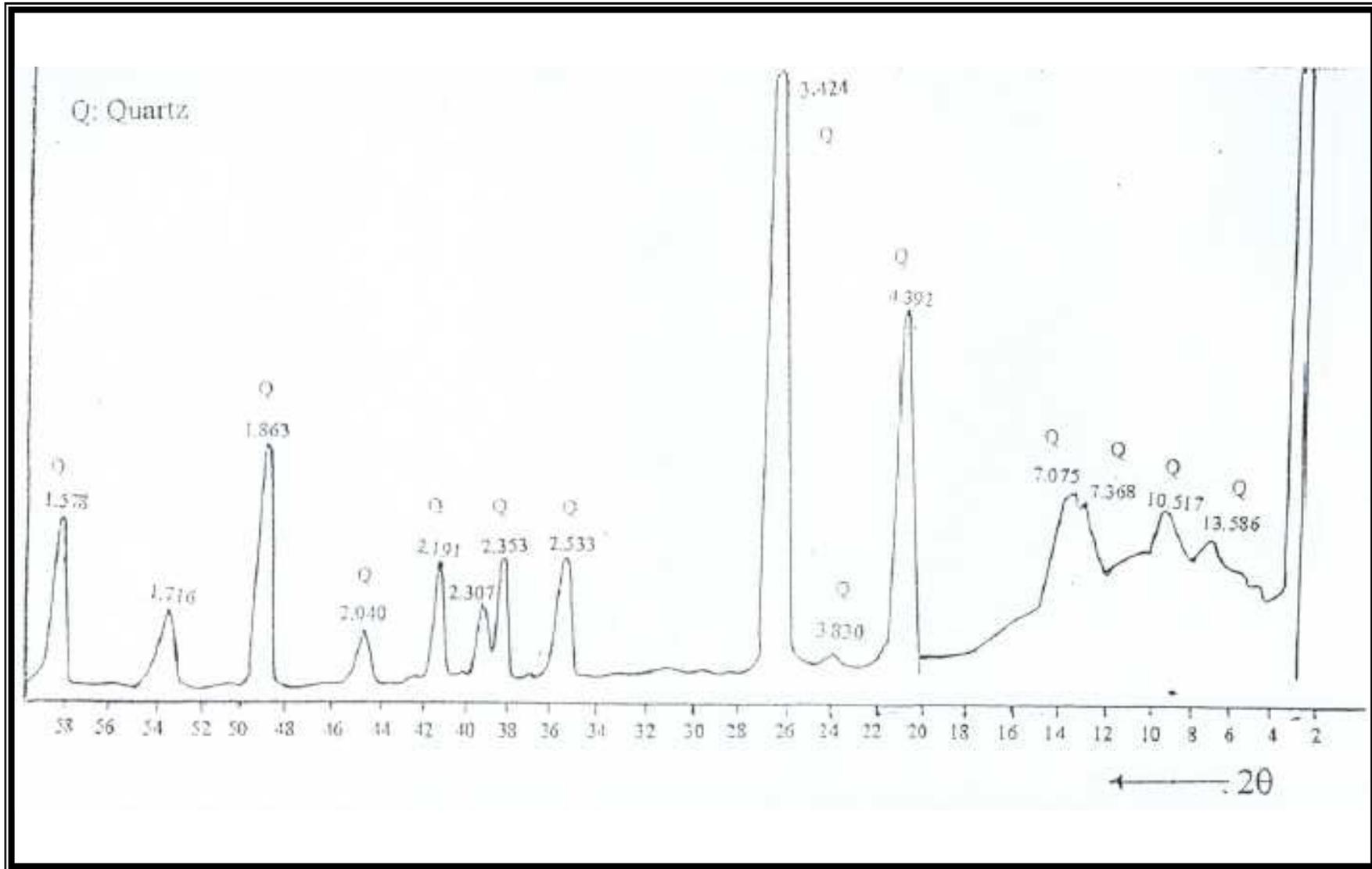


Figure (3 – 3) XRD pattern for Ardhuma sand glass raw material

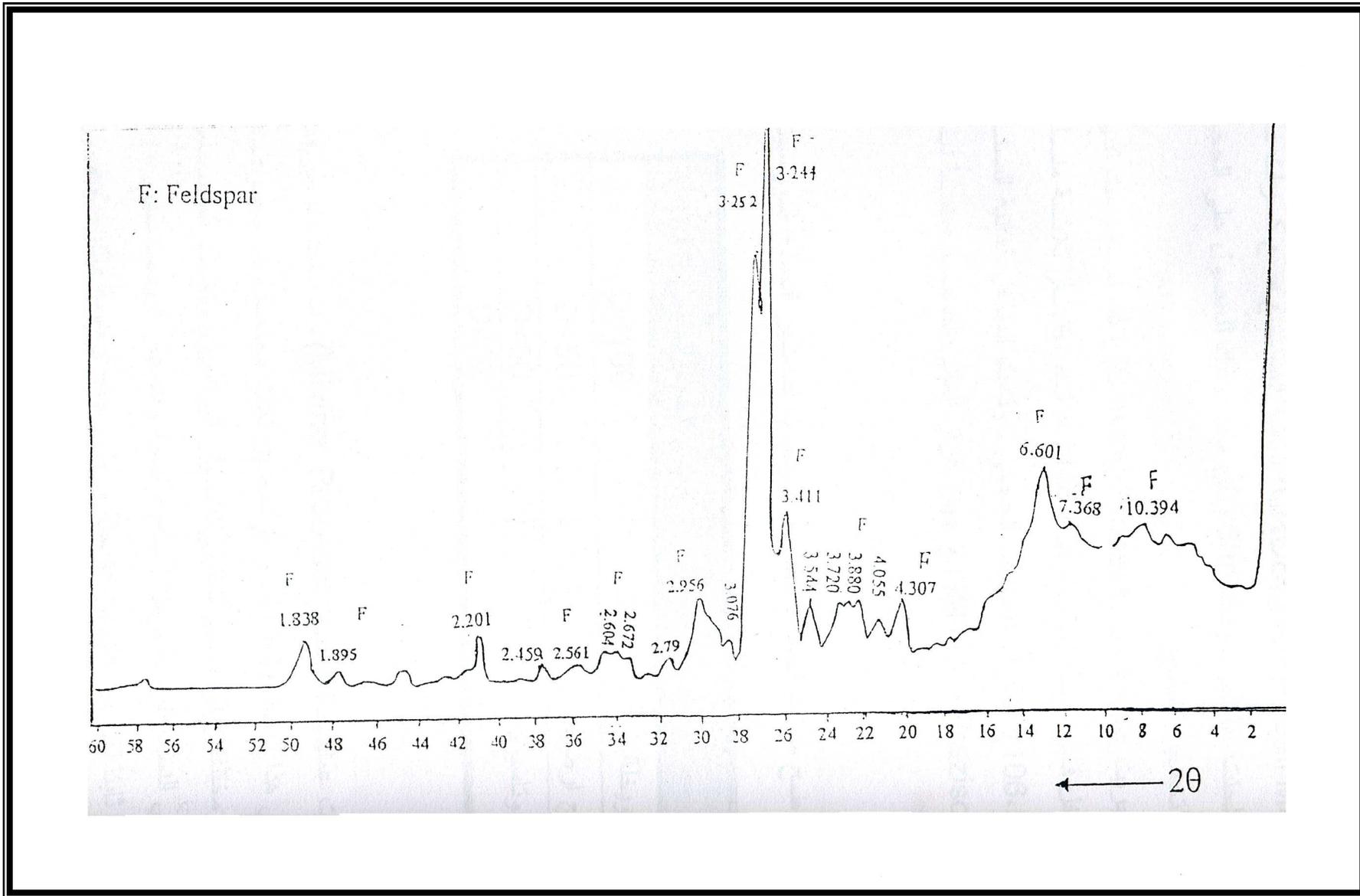


Figure (3-4) XRD pattern for potash feldspar raw material

Kaolin duekhla and Ardhuma sand glass treated by washing them, with HCl (4N), for time duration 24 hours, with stirring, at room temperature. The acid treatment for Kaolin duekhla and Ardhuma sand glass to remove the free iron oxide and other impurities. By this process we can remove all minerals and oxides which can be dissolved in HCl. The effect of this treatment studied qualitatively by IR measurement. The measurements hasbeen made by using (Shimadzu Fourier Transforms infrared model FTIR 8300 (Kyoto, Japan)). The IR spectrum for kaolin raw material in fig. (3-5), fig. (3-6) is for kaolin treated with HCl. The IR spectrum for Ardhuma sand glass raw material is shown in fig. (3-7), while the IR spectrum for Ardhuma sand glass washed by HCl is shown in fig. (3-8). The washed materials then separated using filter papers and under sequence of operation for dilution and filtering until the PH – value become 5. Then the separated materials dried by using furnace (GCA\PRECTIONSIENTIFC, model 16) at temperature range 50–70 °C for 10 hours. The dried powders of Kaolin duekhla and Ardhuma sand glass and the feldspar potash then milled using ball mill of porcelain body, with different sizes of spheres, for 5 hours. The milled powders sieved using different sizes of sieves to obtain powder with particle size give in table (3-2).

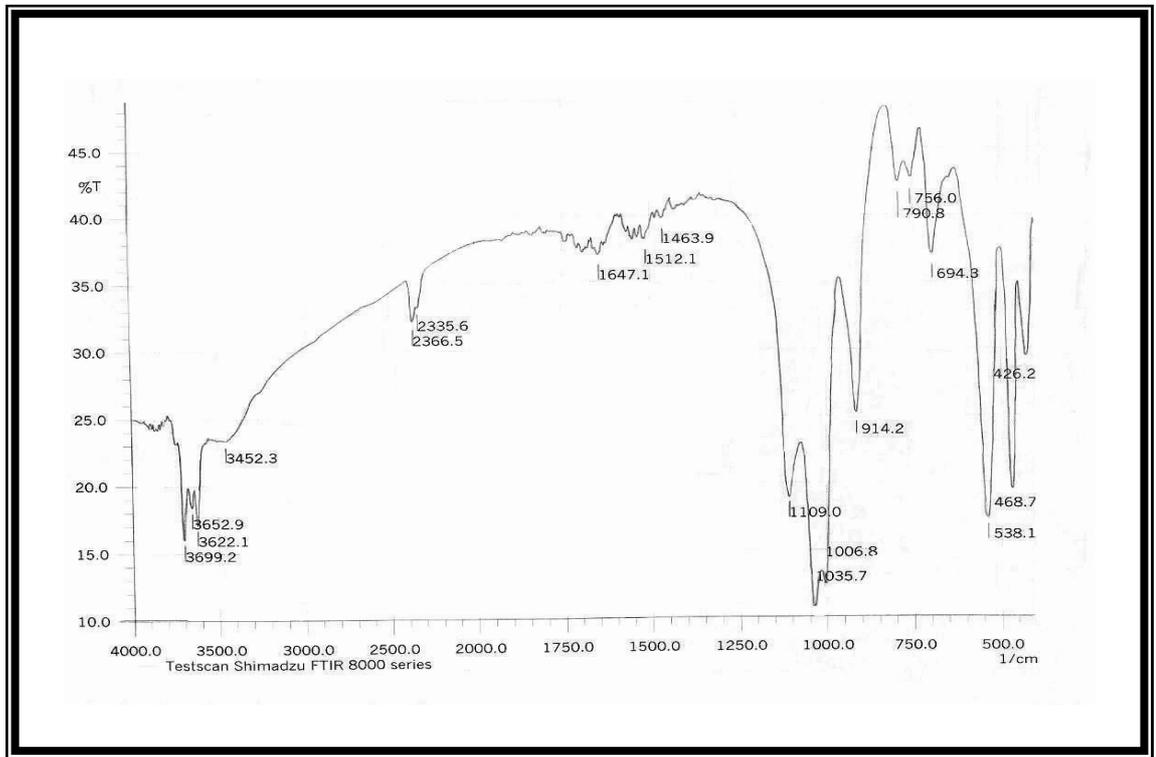


Figure (3–5). IR analysis for kaolin raw material.

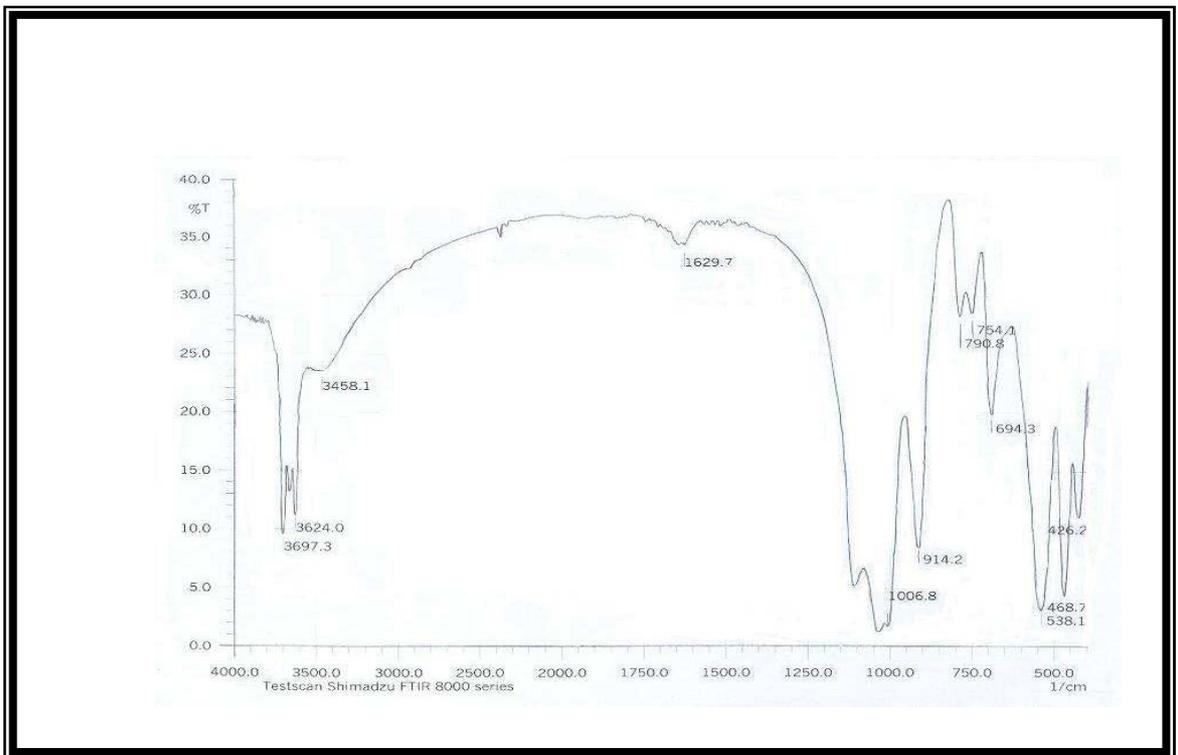


Figure (3–6). IR for analysis for kaolin washed by HCl.

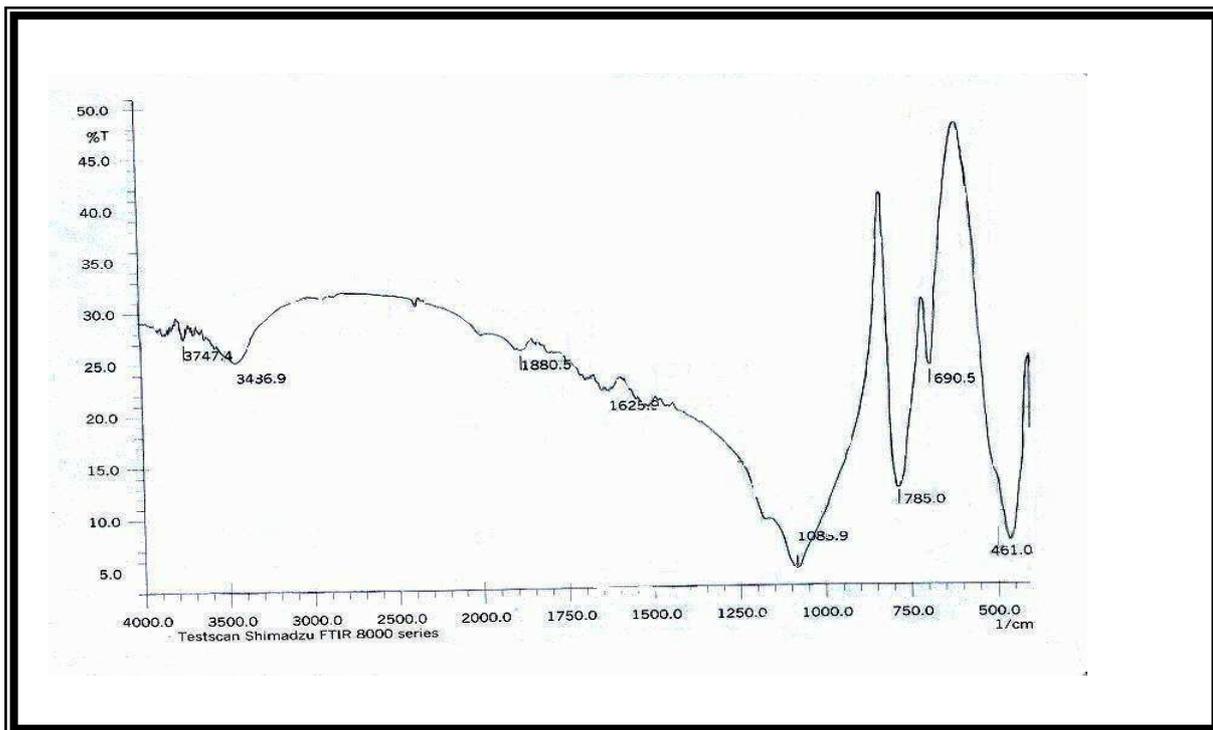


Figure (3-7). IR analysis for Ardhuma sand glass raw material.

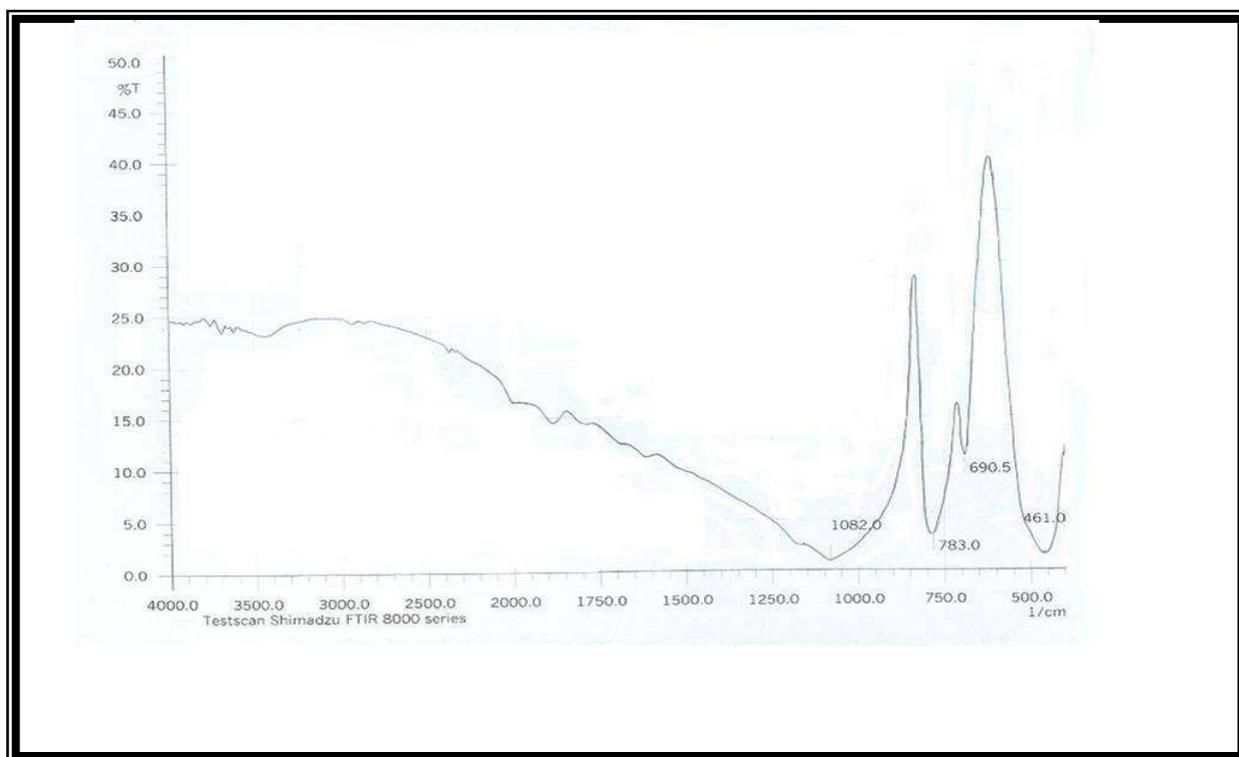


Figure (3-8). IR analysis for Ardhuma sand glass washed by HCl .

**2 - Electrolyte Solution Preparation:** - Electrolyte solution are prepared using  $\text{Na}_2\text{CO}_3$  &  $\text{Na}_2\text{SiO}_3$  by ratio 2 : 1 respectively, from this mixture, we determine the amount of adding distilled water to obtain electrolyte solution with concentration 5 %. So the amount of distill water added to prepared this solution was 114 ml. Mixing 4 gram of  $\text{Na}_2\text{CO}_3$  with 2 gram of  $\text{Na}_2\text{SiO}_3$  and solving these mass weights in 50 ml of distilled water, with continues mixing process for half hour using magnetic stirrer (model (Great BRITAIN, serial 11750)). After that we carried on adding distilled water to obtain a final volume 114 ml. The preparation of electrolyte solutions with the concentrations (1%, 0.7 %, 0.5 %, 0.2 %, and 0.1 %) from mentioned above 5 % done by using dilution equation given by: [59].

$$M_i V_i = M_j V_j \quad \text{----- (3-1)}$$

Where  $M_i$  is the percentage of solution before dilution.

$V_i$  is the volume of solution before dilution.

$M_j$  is the percentage of solution after dilution.

$V_j$  is the volume of solution after dilution.

**3 - Sample Preparation:** - percentages of the washed raw materials are used to prepare the samples; the samples were prepared by using fixed value of percentage washed raw materials, which defined in table (3-2). 2%wt of Zinc Oxide was added to the mixture followed by mixing for 2hour. The final mixture then divided into seven groups related to different percentage of electrolyte solution which is added as given in table (3-2). A polyvanal alcohol binder was prepared and applied with 1%wt for each group. The mixing process was done under heating (80 °C) until it get a slurry form, and then dried at 70°C with continuous mixing for 3 hours, until obtaining agglomerated powders.

Table (3–2).Sample preparation.

| Parameter            |                                  |     |            |                   | Groups            |                |                |                |                |                |                |
|----------------------|----------------------------------|-----|------------|-------------------|-------------------|----------------|----------------|----------------|----------------|----------------|----------------|
|                      |                                  |     |            |                   | M                 | M <sub>1</sub> | M <sub>2</sub> | M <sub>3</sub> | M <sub>4</sub> | M <sub>5</sub> | M <sub>6</sub> |
| Ceramic Component    | Kaolin duekhla                   | Wt% | Washing    | Particle size(μm) | 100% Raw Material |                |                |                |                |                |                |
|                      | Ardhuma sand                     | 45% | 4N HCl     | < 100             |                   |                |                |                |                |                |                |
|                      | Feldspar                         | 25% | 4N HCl     | < 75              |                   |                |                |                |                |                |                |
| Electrolyte Solution | Component                        |     | Percentage |                   |                   |                |                |                |                |                |                |
|                      | Na <sub>2</sub> CO <sub>3</sub>  |     | 2          |                   | Zero              | Zero           | 1%             | 0.7%           | 0.5%           | 0.2%           | 0.1%           |
|                      | Na <sub>2</sub> SiO <sub>3</sub> |     | 1          |                   |                   |                |                |                |                |                |                |
| Mineralizer          | ZnO                              |     |            |                   | Zero              | 2%             | 2%             | 2%             | 2%             | 2%             | 2%             |
| Binder               | PVA                              |     |            |                   | 1%                | 1%             | 1%             | 1%             | 1%             | 1%             | 1%             |

The final powder was milled for about one hour, and then sieved using a sieve of size 250 μm. The sieved powders then pressed by using press (model (38888.4D10A00, made in USA)), with pressure 7MPa as disc form of diameter 30 mm and 3mm thickness. These samples were dried in a furnace at a temperature 70 °C for two hours. The prepared samples burnt by a furnace (model (Hi 62, Ti7, 1700, Nabertherm)) using different temperatures 1250, 1300, and 1350 °C, with sintering time 2 hr. and sintering rate 100 °C/hr.

### **3.3 Measurements:**

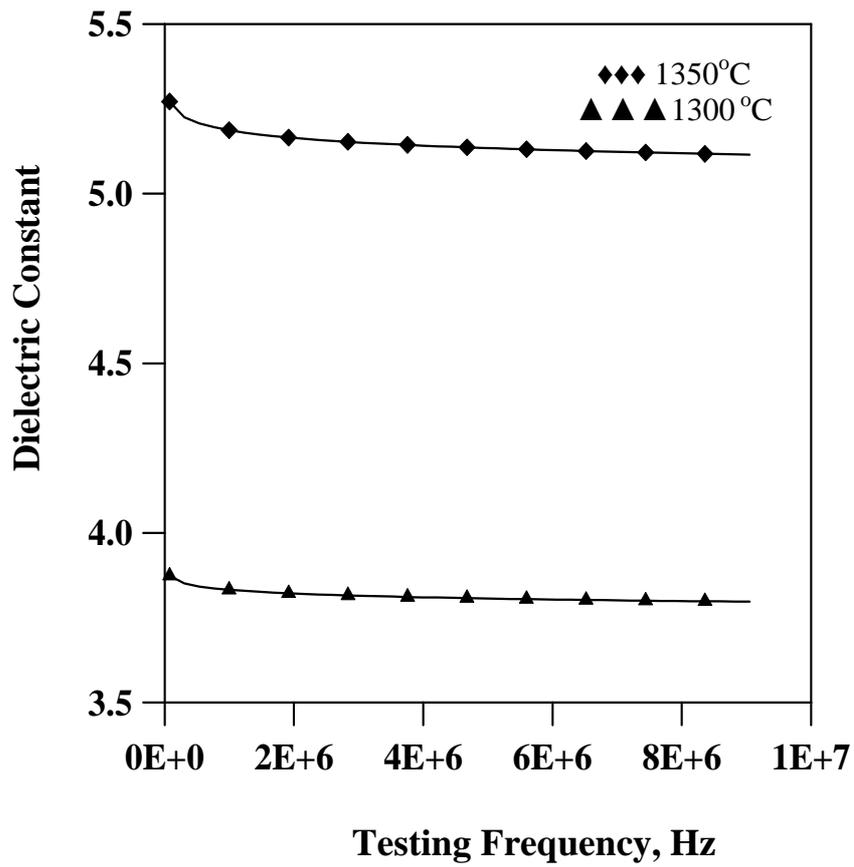
#### **3.3.1 Dielectric Properties Measurements**

The capacitance  $C_p$  and  $\tan \delta$  of the sample are measured by LCR meter (model (Agilent 4294A, Parcition impedance analysis, 40Hz to 110Hz)).

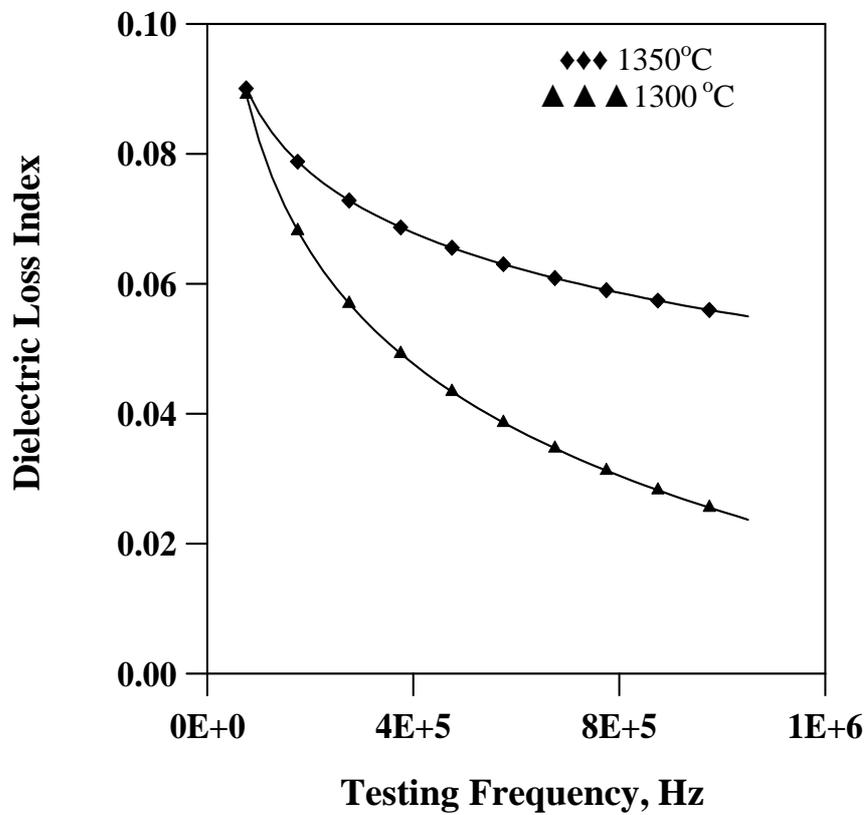
Dielectric constant are calculated by using equation (1–4), and dielectric loss index is calculated by using equation (1–13).The results are plotted against applied frequency, as a function of two sintering temperatures(1300°C,1350 °C). The measuring frequency range is from 40Hz to 1 MHz; as table (3–3) which is sufficient to satisfy the requirement for the dielectric measurements in the present work. The behavior of dielectric constant and dielectric loss index were taken of each applied frequency as shown in figures from (3–9) to (3–15).

Table (3 – 3) Dielectric properties of Samples.

| Sintering Temperatures °C |                           |             | 1300                         |                                | 1350                         |                                |
|---------------------------|---------------------------|-------------|------------------------------|--------------------------------|------------------------------|--------------------------------|
| Group No.                 | Condition                 |             | Dielectric Constant at 1 MHz | Dielectric Loss Index at 1 MHz | Dielectric Constant at 1 MHz | Dielectric Loss Index at 1 MHz |
|                           | Electrolyte Concentration | Mineralizer |                              |                                |                              |                                |
| M                         | —                         | —           | 3.859                        | 0.0272                         | 5.164                        | 0.0692                         |
| M <sub>1</sub>            | —                         | 2%ZnO       | 4.533                        | 0.019                          | 5.86                         | 0.0424                         |
| M <sub>2</sub>            | 1%                        | 2%ZnO       | 3.55                         | 0.0039                         | 4.53                         | 0.011                          |
| M <sub>3</sub>            | 0.7%                      | 2%ZnO       | 4.614                        | 0.0325                         | 4.753                        | 0.0269                         |
| M <sub>4</sub>            | 0.5%                      | 2%ZnO       | 4.475                        | 0.01                           | 4.3747                       | 0.0335                         |
| M <sub>5</sub>            | 0.2%                      | 2%ZnO       | 4.7201                       | 0.0169                         | 5.7895                       | 0.0354                         |
| M <sub>6</sub>            | 0.1%                      | 2%ZnO       | 4.89                         | 0.0335                         | 5.81                         | 0.0533                         |

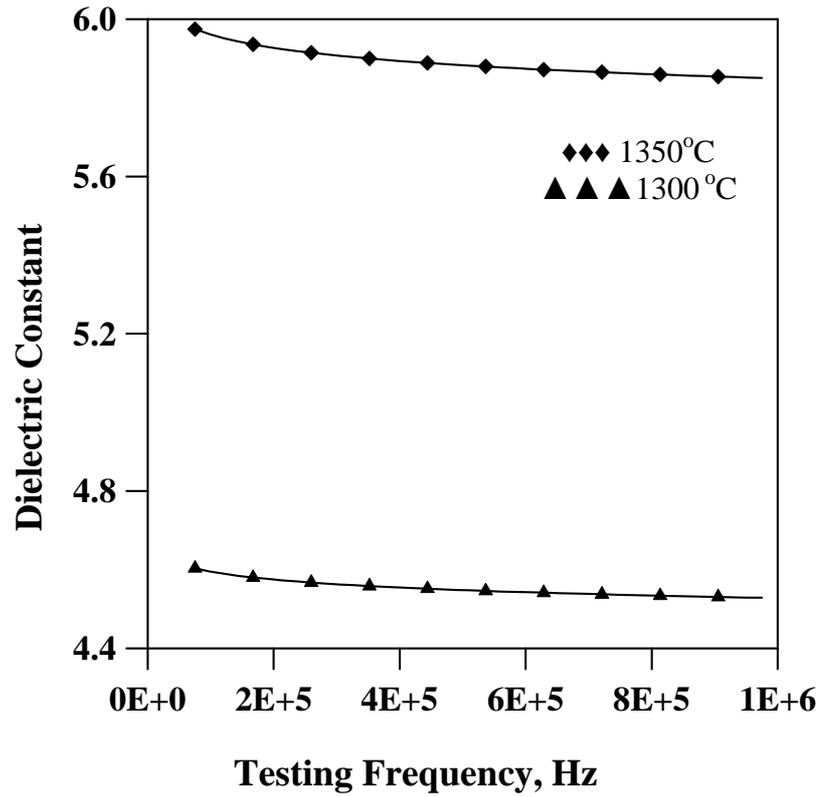


(a)

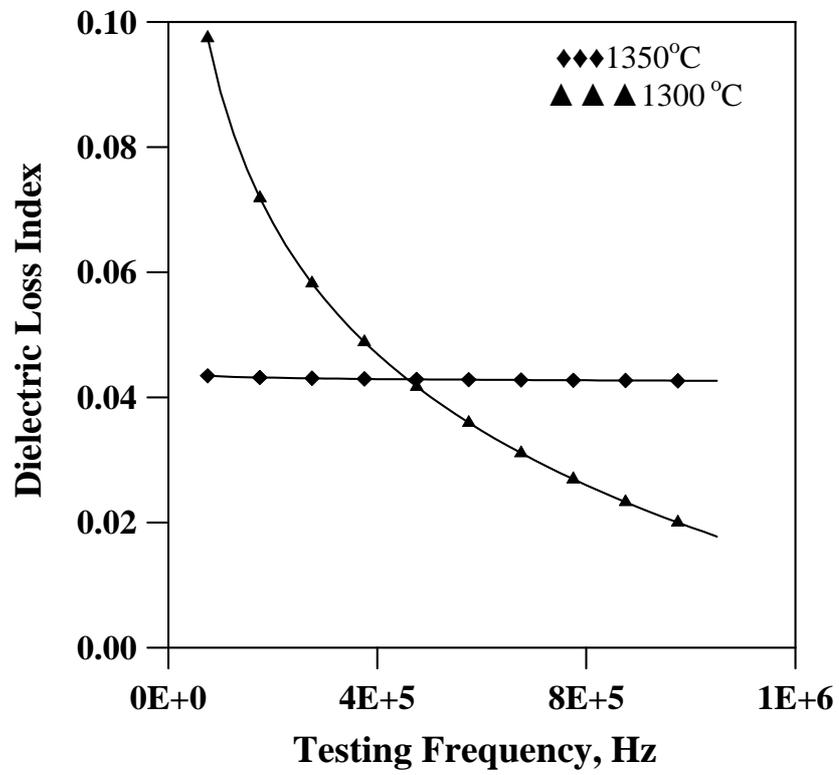


(b)

Figure (3–9). (a) Dielectric Constant, (b) Dielectric Loss Index; for group M

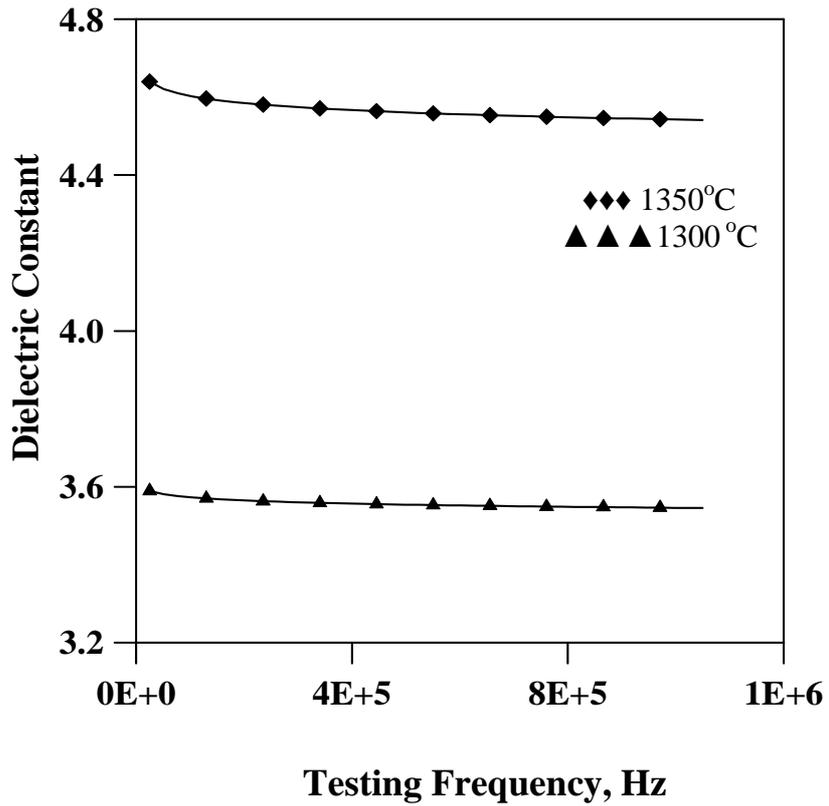


(a)

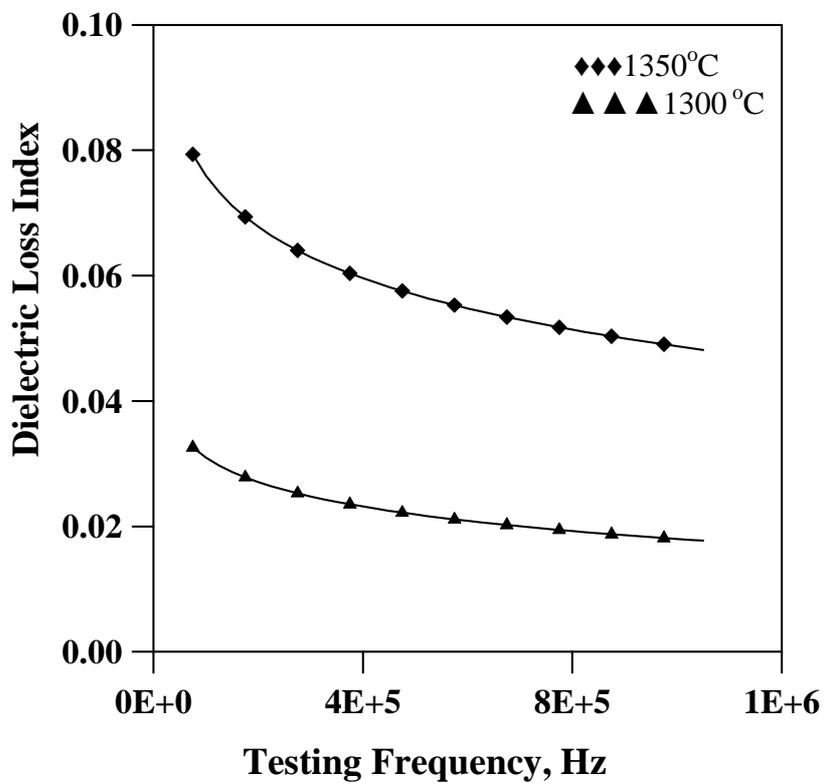


(b)

Figure (3-10). (a) Dielectric Constant, (b) Dielectric Loss Index; for group  $M_1$ .

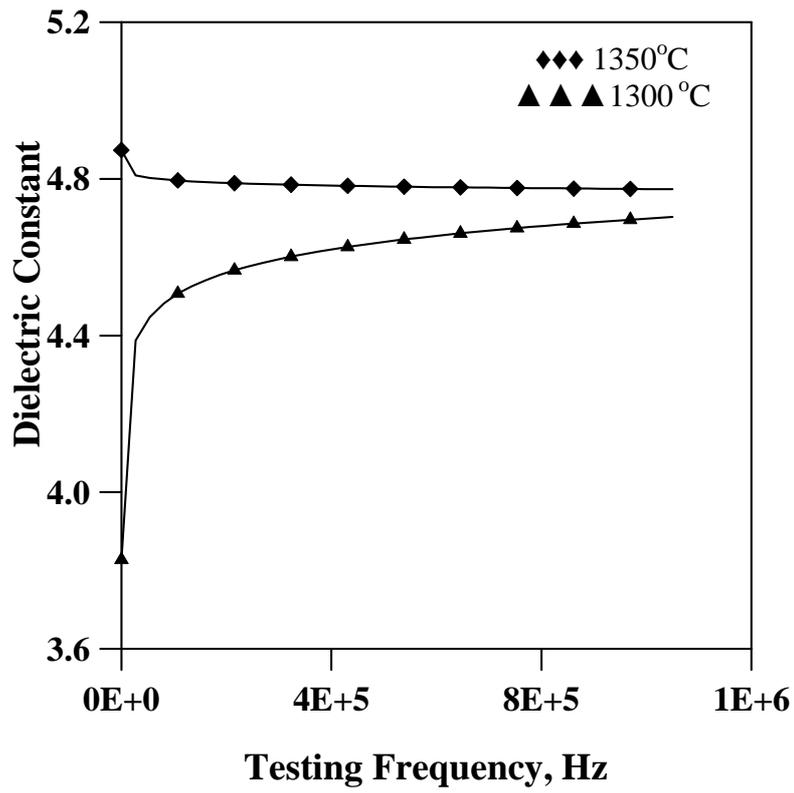


(a)

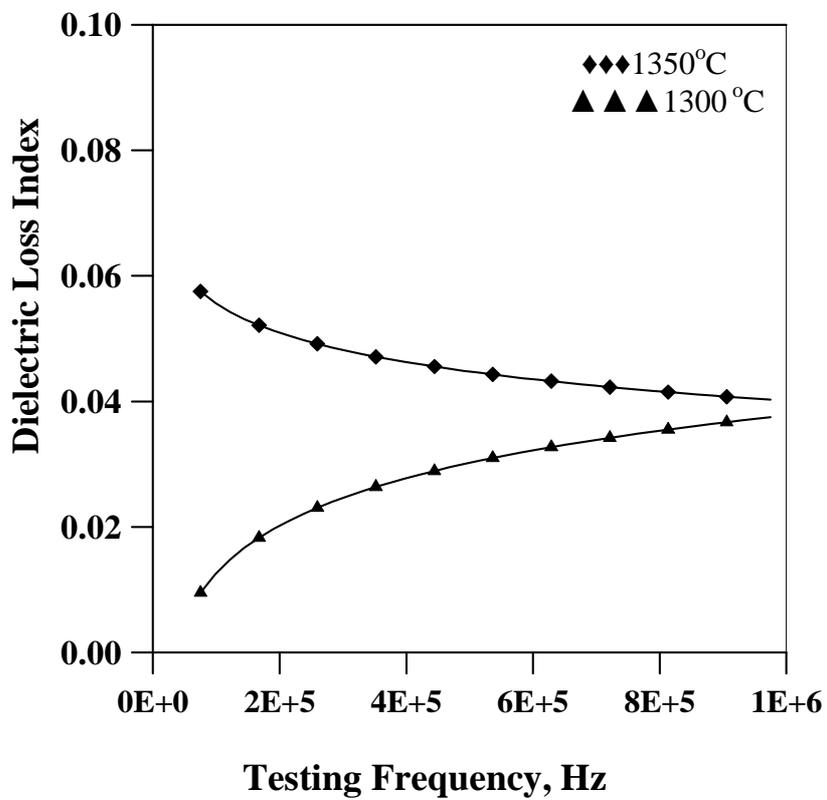


(b)

Figure (3–11). (a) Dielectric Constant, (b) Dielectric Loss Index; for group  $M_2$ .

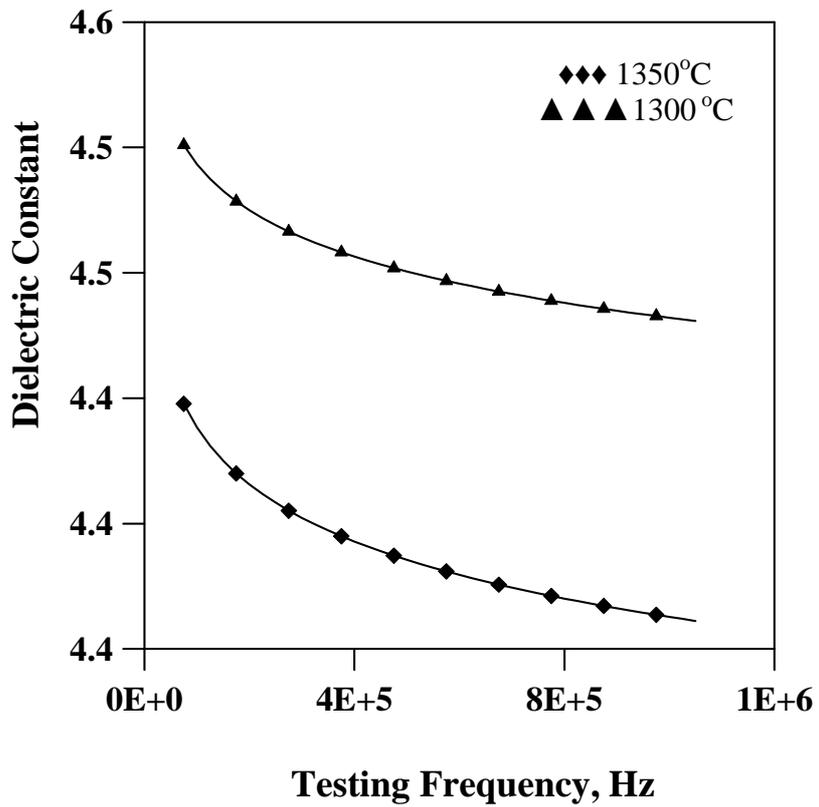


(a)

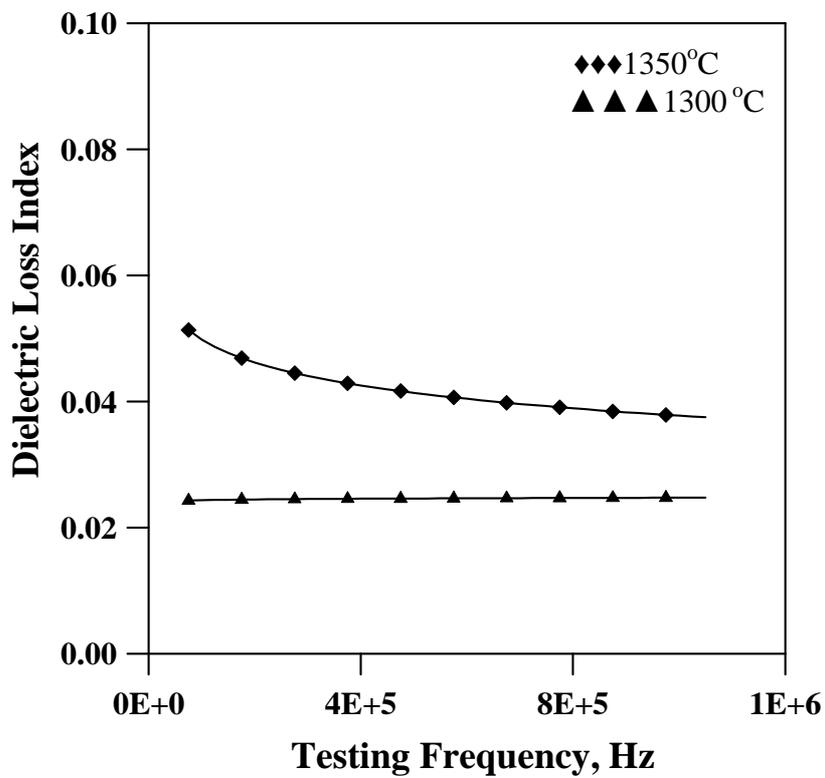


(b)

Figure (3–12). (a) Dielectric Constant, (b) Dielectric Loss Index; for group  $M_3$ .

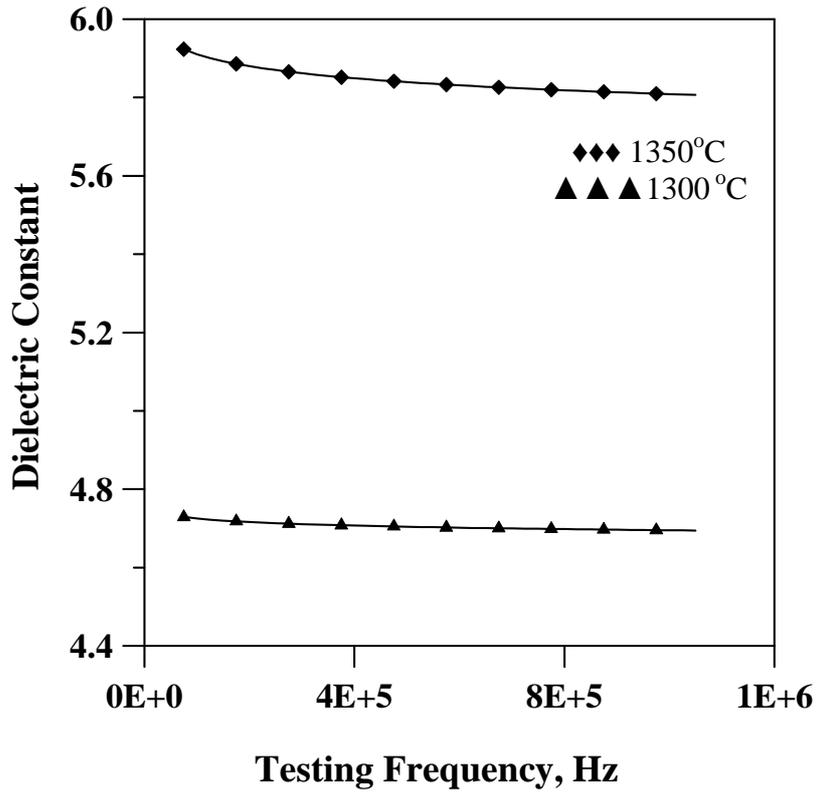


(a)

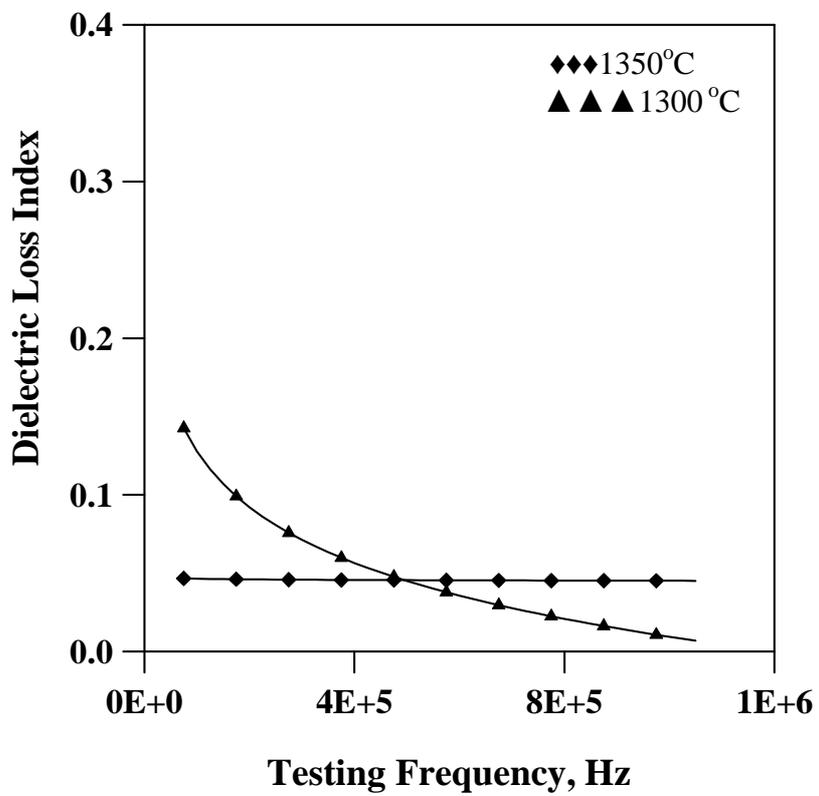


(b)

Figure (3-13). (a) Dielectric Constant, (b) Dielectric Loss Index; for group  $M_4$ .

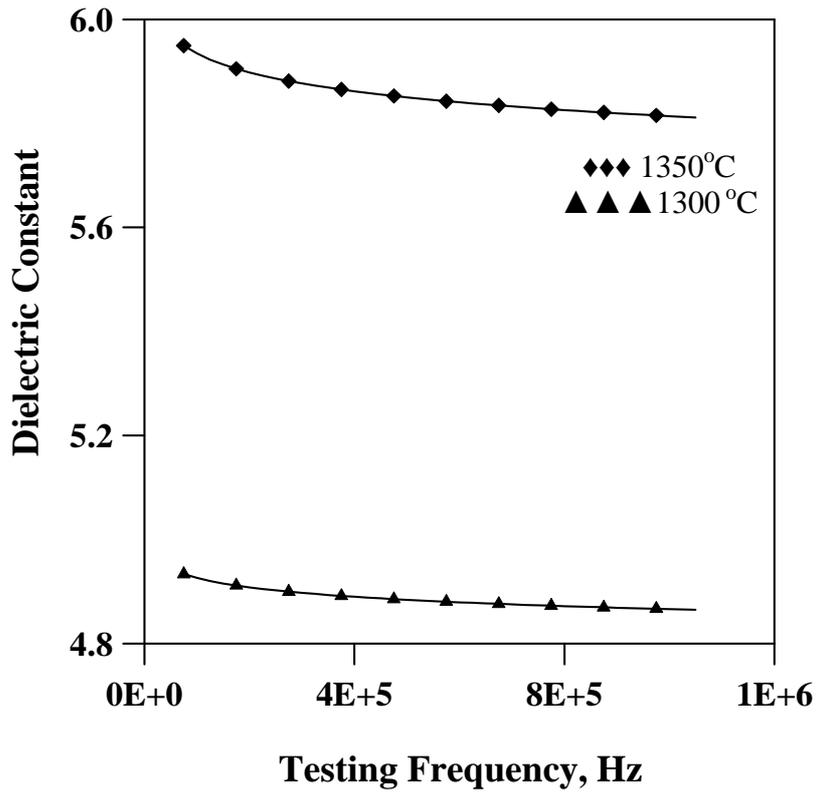


(a)

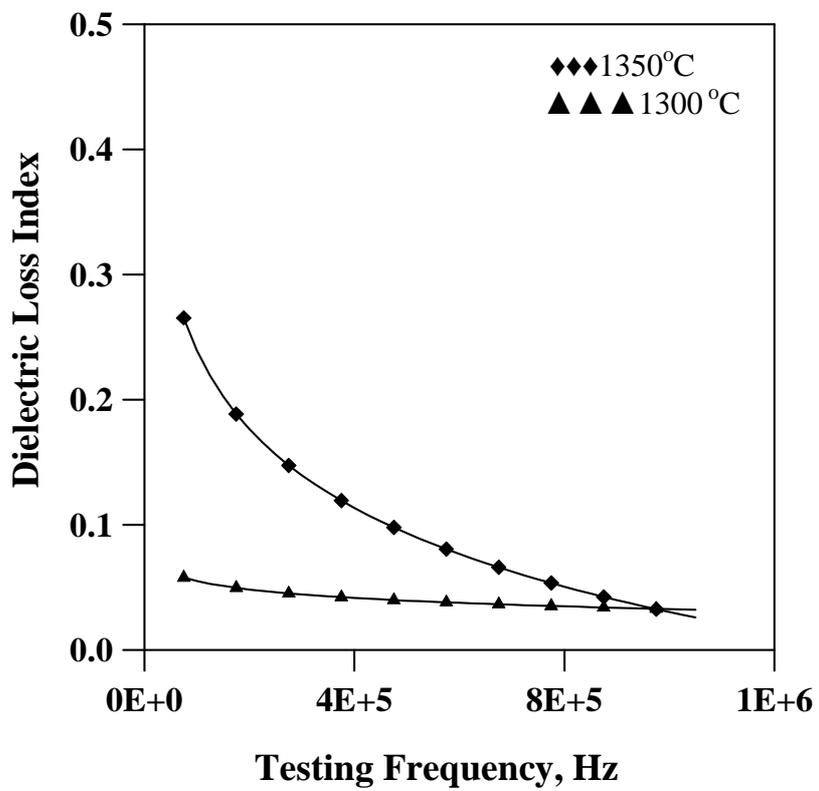


(b)

Figure (3-14). (a) Dielectric Constant, (b) Dielectric Loss Index; for group  $M_5$ .



(a)



(b)

Figure (3-15). (a) Dielectric Constant, (b) Dielectric Loss Index; for group  $M_6$ .

### 3.3.2 Physical Properties Measurements

The thermal conductivity was measured for all the sample by using less disc discussed in section 1.3.2, the diameter of the prepared samples  $r_d=27\text{mm}$ ,  $I=0.25\text{A}$ , and  $V= 6$  volts. The results are given in table (3–3).

*Table (3–3). The thermal conductivity of samples for different sintering temperatures.*

| Thermal conductivity value (w / m . °C)                                  |                           |                          |          |          |
|--|---------------------------|--------------------------|----------|----------|
| Group No.  | Electrolyte concentration | Sintering temperature °C |          |          |
|  |                           | 1250                     | 1300     | 1350     |
| M  | —                         | 0.108490                 | 0.177611 | 0.225793 |
| M <sub>1</sub>   | —                         | 0.137455                 | 0.164654 | 0.129662 |
| M <sub>2</sub>   | 1%                        | 0.199339                 | 0.184806 | 0.179532 |
| M <sub>3</sub>   | 0.7%                      | 0.255555                 | 0.209383 | 0.213826 |
| M <sub>4</sub>   | 0.5%                      | 0.213739                 | 0.156372 | 0.156605 |
| M <sub>5</sub>   | 0.2%                      | 0.218960                 | 0.176473 | 0.230401 |
| M <sub>6</sub>   | 0.1%                      | 0.186692                 | 0.169690 | 0.205713 |
| Standard value of Porcelain= $0.25 \times 10^{-3}$ (w / m . °C) Ref.[11] |                           |                          |          |          |

### 3.3.3 Mechanical properties Measurements:

The Compressive Strength for all samples was measured by using (Material Testing Unit System) .The results are given in table (3- 4).

*Table (3-4). The Compressive Strength of samples for different value of sintering temperature.*

| Compressive Strength(MPa)                            |                           |                          |        |         |
|--|---------------------------|--------------------------|--------|---------|
| Group No.  | Electrolyte concentration | Sintering temperature °C |        |         |
|  |                           | 1250                     | 1300   | 1350    |
| M  | —                         | 11.0117                  | 22.425 | 30.7211 |
| M <sub>1</sub>                                       | —                         | 14.015                   | 18.226 | 35.060  |
| M <sub>2</sub>                                       | 1%                        | 7.539                    | 23.791 | 42.230  |
| M <sub>3</sub>                                       | 0.7%                      | 11.052                   | 21.528 | 40.460  |
| M <sub>4</sub>                                       | 0.5%                      | 1.1317                   | 27.374 | 64.6844 |
| M <sub>5</sub>                                       | 0.2%                      | 11.823                   | 31.162 | 52.0462 |
| M <sub>6</sub>                                       | 0.1%                      | 15.310                   | 12.696 | 48.3166 |
| Standard value of Porcelain=5.1 to 44 (MPa). Ref.[8] |                           |                          |        |         |

## Chapter Four

### Discussion of Results, Conclusions, & Future Work

#### 4.1 Raw Material Characterization:-

Infrared (IR) curves for kaolin duekhla and kaolin duekhla which was washed by HCl are shown in fig (3–5) and fig (3–6) this resulting from the present investigation are listed in table (4–1) are comparable with data listed by Marel [60]. These measurements were given in table (4–1) reveals the following:-

1-The major these bands of kaolin Duekhla raw material (particle size < 100µm) are 3699.2, 3622.1 and 3652.9 cm<sup>-1</sup>.

2- Washed kaolin Duekhla by hydrochloric acid affects the intensity of major bands were lowered and the band 3652.9 cm<sup>-1</sup> was disappeared.

*Table (4– 1) Absorption bands in kaolin Duekhla..*

| material                        | bands                      |                  |  |
|---------------------------------|----------------------------|------------------|--|
|                                 | O – H                      | H <sub>2</sub> O | Minerals   |
| kaolin duekhla                  | 3699.2<br>3622.1<br>3652.9 | 1512.1           | 1463.9<br>1109.0<br>1035.7- 1006.8<br>914.2<br>790.8- 756.0<br>694.3 |
| Kaolin duekhla treated with HCl | 3697.3<br>3624.0<br>—      | —                | —<br>—<br>1006.8<br>914.2<br>790.8 - 754.1<br>694.3                  |

Table (4–2) Absorption bands in Aruthma sand glass.

| Material                            | Bands   |                  |          |
|-------------------------------------|---------|------------------|----------|
|                                     | O – H   | H <sub>2</sub> O | Minerals |
| Aruthma Sand Glass                  | 3747.42 | 1625.9           | 783.0    |
|                                     | 3436.9  |                  | 690.5    |
| Aruthma Sand Glass<br>Washed by HCl | –       | –                | 785.0    |
|                                     | –       |                  | 690.5    |

Infrared (IR) curves for Aruthma Sand Glass and Aruthma Sand Glass washed by HCl are shown in fig (3–6) and fig (3–7) this resulting from the present investigation are listed in table (4–2) which are comparable with data listed by Marel [60]. From measurements were given in table (4–2) it can be noticed that:-

- 1- The major two bonds of Aruthma sand glass raw material (particle size <math><75\mu\text{m}</math>) are 3747.42 and 3436.9 $\text{cm}^{-1}$ .
- 2- The effects of washed by HCl; is the absence of (O–H) absorption bonds and H<sub>2</sub>O band.

## **4.2 Discussion of the Results:-**

Three types of measurements were carried out on the samples of groups specified in this study. These measurements are regarded as controlling parameters and specified properties for ceramic body, which are dielectric properties, thermal properties, mechanical properties.

The dielectric properties are dielectric constant & dielectric loss index. The definition and mechanism are defined in previous chapter. The results are shown by the figures (3–9) to (3–15), which explain the behavior of dielectric constant and dielectric loss index as a function of frequency, and as a function of sintering temperature. From fig. (3–9) which is note the change of dielectric constant and dielectric loss index for sample of group M which is haven't any addition, sintering at temperatures (1300 and 1350 °C) and by comparing with result in fig. (3–10) which illustrated the change of dielectric constant and dielectric loss index with testing frequency for sample of group M<sub>1</sub> which is containing 2%ZnO and sintering at temperatures 1300 and 1350 °C, it is found these values of dielectric constant and dielectric loss index of group M<sub>1</sub> is grater than values of M in both temperatures, this returns to the effect of ZnO to improved the electrical properties. And by comparing the result from fig.(3–10) with results in fig.(3–11) which is described the change of dielectric constant and dielectric loss index with testing frequency of the sample of group M<sub>2</sub> which is contain 1% concentration of electrolyte solution, it is notice these values of dielectric constant and dielectric loss index less than values of group M<sub>1</sub> that has 2%ZnO. Figures (3–12 ) to (3–13) for group M<sub>3</sub>, M<sub>4</sub>, shown a more stable change for dielectric constant and dielectric loss index as frequency increases, which can regarded that using 0.2–0.5% concentration of electrolyte solution may gives uniform glassing phase under firing condition of 1350 °C .



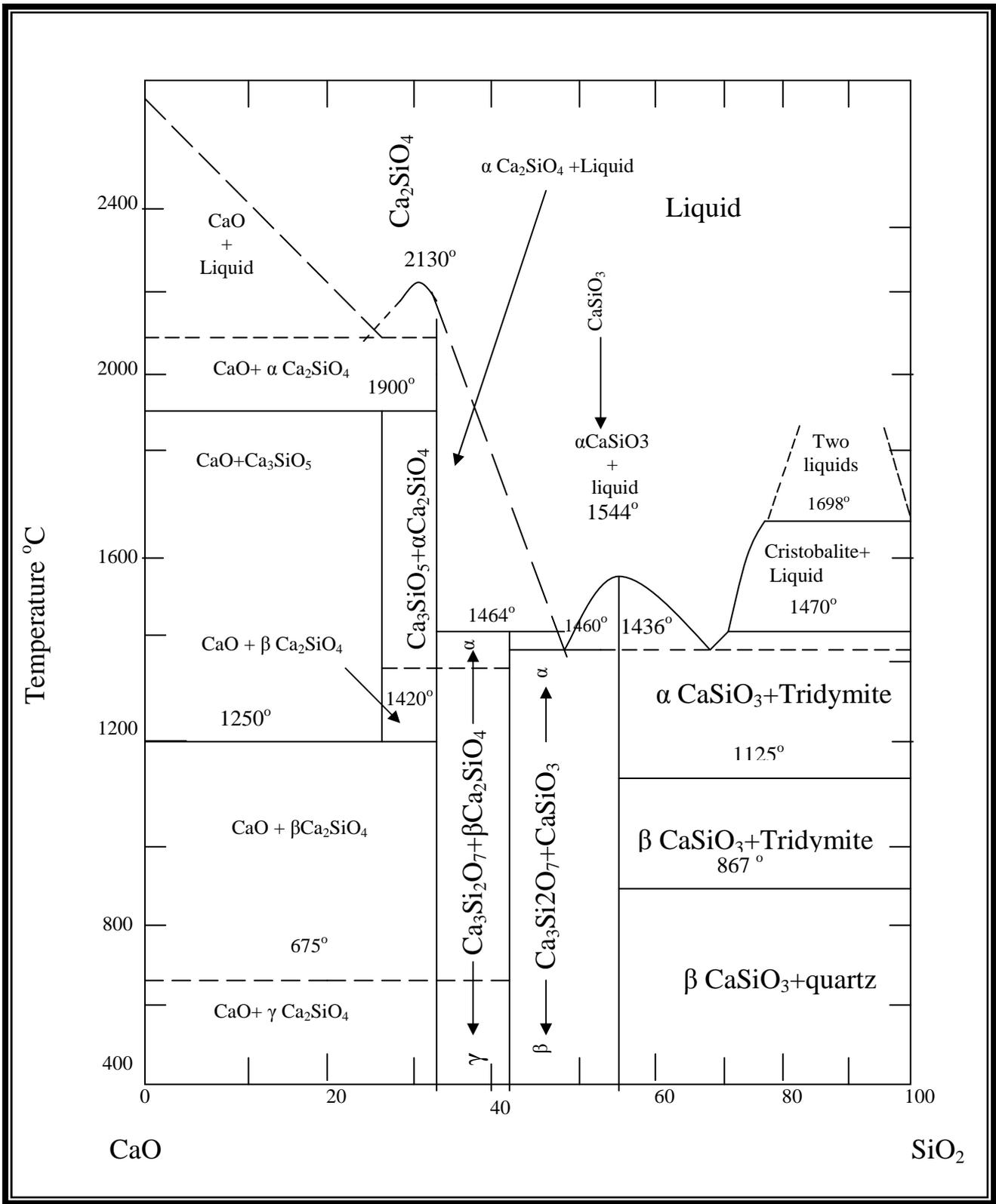


Figure (4-2) Phase equilibrium diagram of CaO – SiO<sub>2</sub> [6].

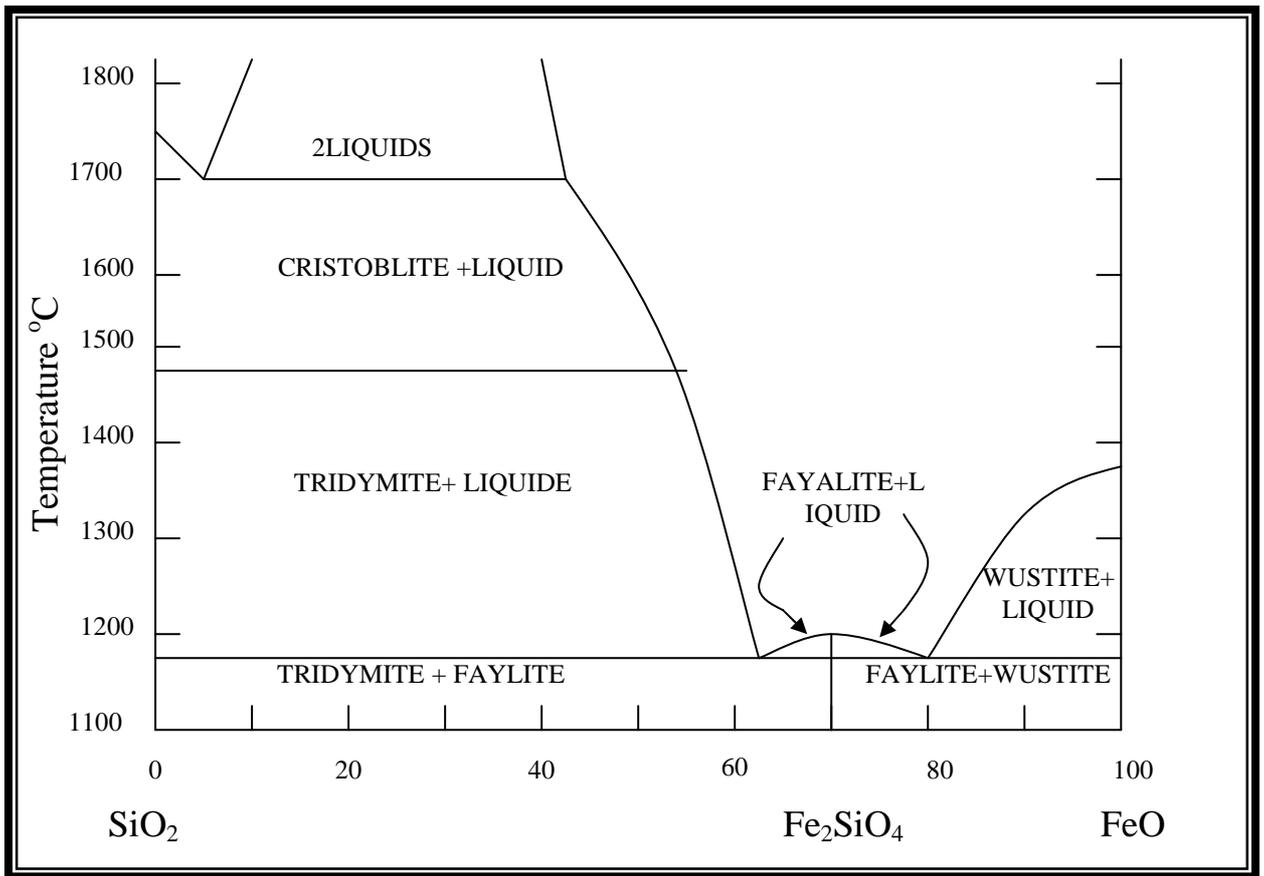


Figure (4- 3) The Iron oxide / Silica phase diagram[6].

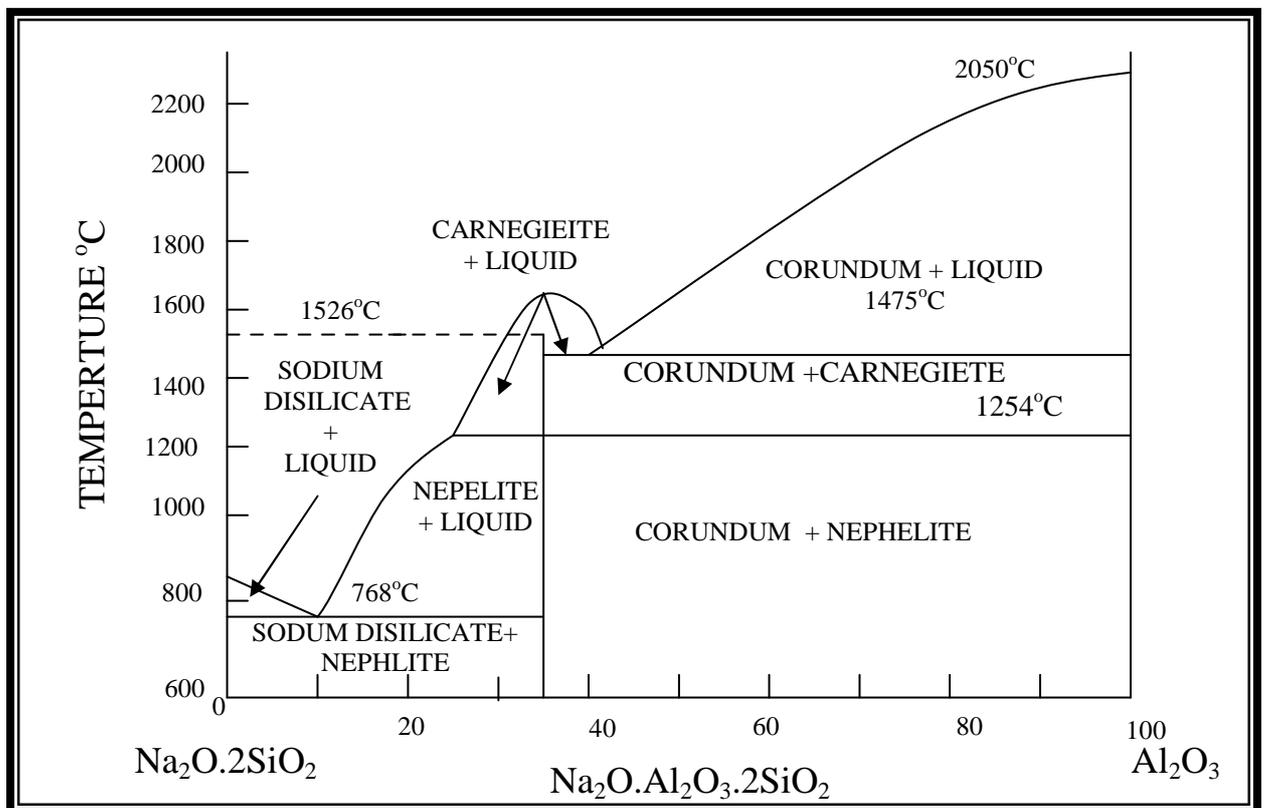


Figure (4-4) The phase relationships between Sodium Disilicate and Corundum[6].

As shown from these figures, and by the comparison with the percentage contribution in the each group (i.e.  $\text{Al}_2\text{O}_3\%$ ,  $\text{SiO}_2\%$ ,  $\text{TiO}_2\%$ ,  $\text{MgO}\%$ ,  $\text{Na}_2\text{O}\%$ ,  $\text{K}_2\text{O}\%$ ,  $\text{CaO}\%$  etc.), which may defined in table (4–3).

*Table (4–3). Chemical composition of each group.*

| Group No.    | Percentage of oxides |                         |                         |                |              |              |               |                       |                      |       |
|--------------|----------------------|-------------------------|-------------------------|----------------|--------------|--------------|---------------|-----------------------|----------------------|-------|
|              | $\text{SiO}_2$       | $\text{Fe}_2\text{O}_3$ | $\text{Al}_2\text{O}_3$ | $\text{TiO}_2$ | $\text{CaO}$ | $\text{MgO}$ | $\text{SO}_3$ | $\text{Na}_2\text{O}$ | $\text{K}_2\text{O}$ | L.O.1 |
| M            | 68.78                | 0.648                   | 19.925                  | 0.054          | 0.8955       | 0.54175      | 0.2025        | 1.02                  | 2.5                  | 5.858 |
| $\text{M}_1$ | 68.78                | 0.648                   | 19.925                  | 0.054          | 0.8955       | 0.54175      | 0.2025        | 1.02                  | 2.5                  | 5.858 |
| $\text{M}_2$ | 69.78                | 0.648                   | 19.925                  | 0.054          | 0.8955       | 0.54175      | 0.2025        | 2.02                  | 2.5                  | 5.858 |
| $\text{M}_3$ | 69.4                 | 0.648                   | 19.925                  | 0.054          | 0.8955       | 0.54175      | 0.2025        | 1.72                  | 2.5                  | 5.858 |
| $\text{M}_4$ | 69.2                 | 0.648                   | 19.925                  | 0.054          | 0.8955       | 0.54175      | 0.2025        | 1.52                  | 2.5                  | 5.858 |
| $\text{M}_5$ | 68.9                 | 0.648                   | 19.925                  | 0.054          | 0.8955       | 0.54175      | 0.2025        | 1.22                  | 2.5                  | 5.858 |
| $\text{M}_6$ | 68.8                 | 0.648                   | 19.925                  | 0.054          | 0.8955       | 0.54175      | 0.2025        | 1.12                  | 2.5                  | 5.858 |

Now, we can say the above reaction have a great effect by the change of the properties with concentration of electrolyte solution as figures (4–5) and (4–6).

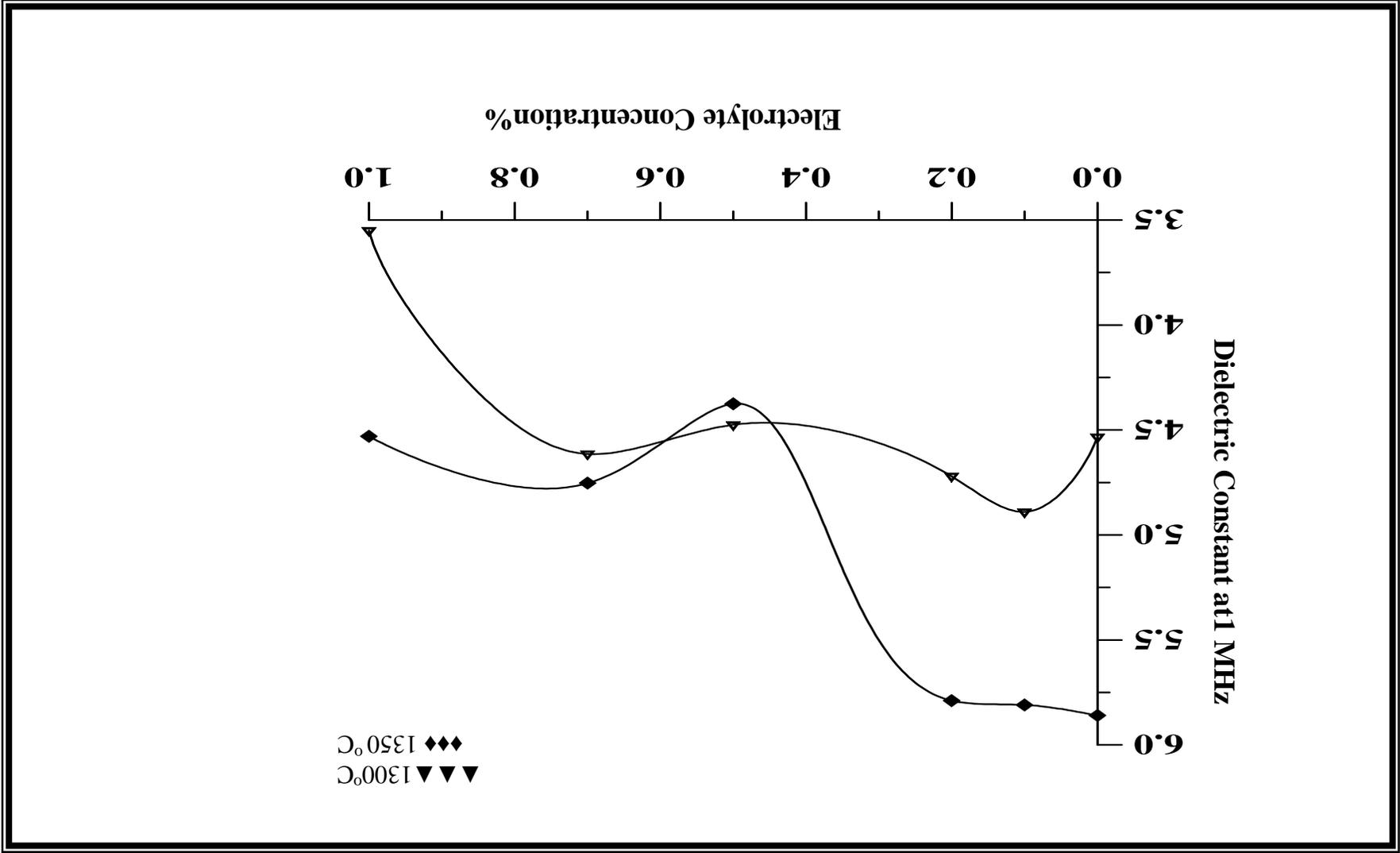


Figure (4-5). The change of dielectric constant with concentration of electrolyte solution.

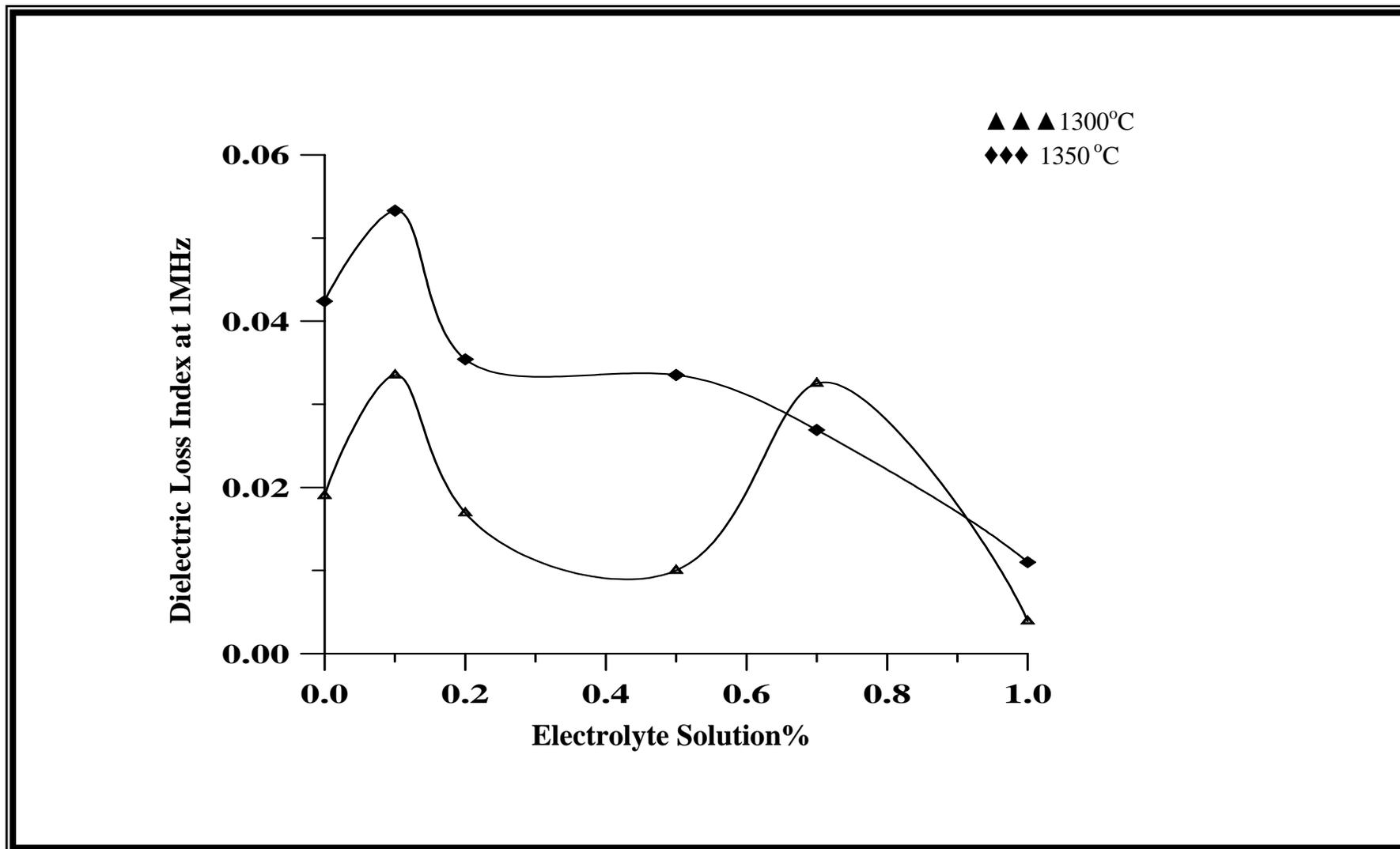


Figure (4-6). The change of dielectric loss index with concentration of electrolyte

Thermal conductivity of porcelain consider low, but the thermal conductivity of  $\text{Al}_2\text{O}_3$  is consider high at (100 °C). On the practical approach one can show that the behavior of thermal conductivity as a function to the concentration of electrolyte solution is non – linear, but have the similar behavior of change with the concentration increases, this because of the effect of many parameters come from structural formation through heat treatment under the effect of electrolyte solution. The effect can not be studied in this investigation because it needs the advanced techniques and analysis which regarded as a study for future work (i.e. chemical composition, porosity, thermal conductivity at high temperatures.)

The results of thermal conductivity are given in table (3–3). It should be that noticed the thermal conductivity increase with the decrease of percentage of electrolyte concentration, and the samples that sintered at high temperature (1350°C) show high value of thermal conductivity.

Groups M and  $M_1$  have result of thermal conductivity less than other groups ( $M_2, M_3, M_4, M_5, M_6$ ) that has concentration of electrolyte solution as show in fig.(4–7).

The study concern with the selection the properties value satisfied as an optimization with other properties, so that the best selected regions of thermal conductivity are in the range 0.2– 0.5% concentration of electrolyte solution.

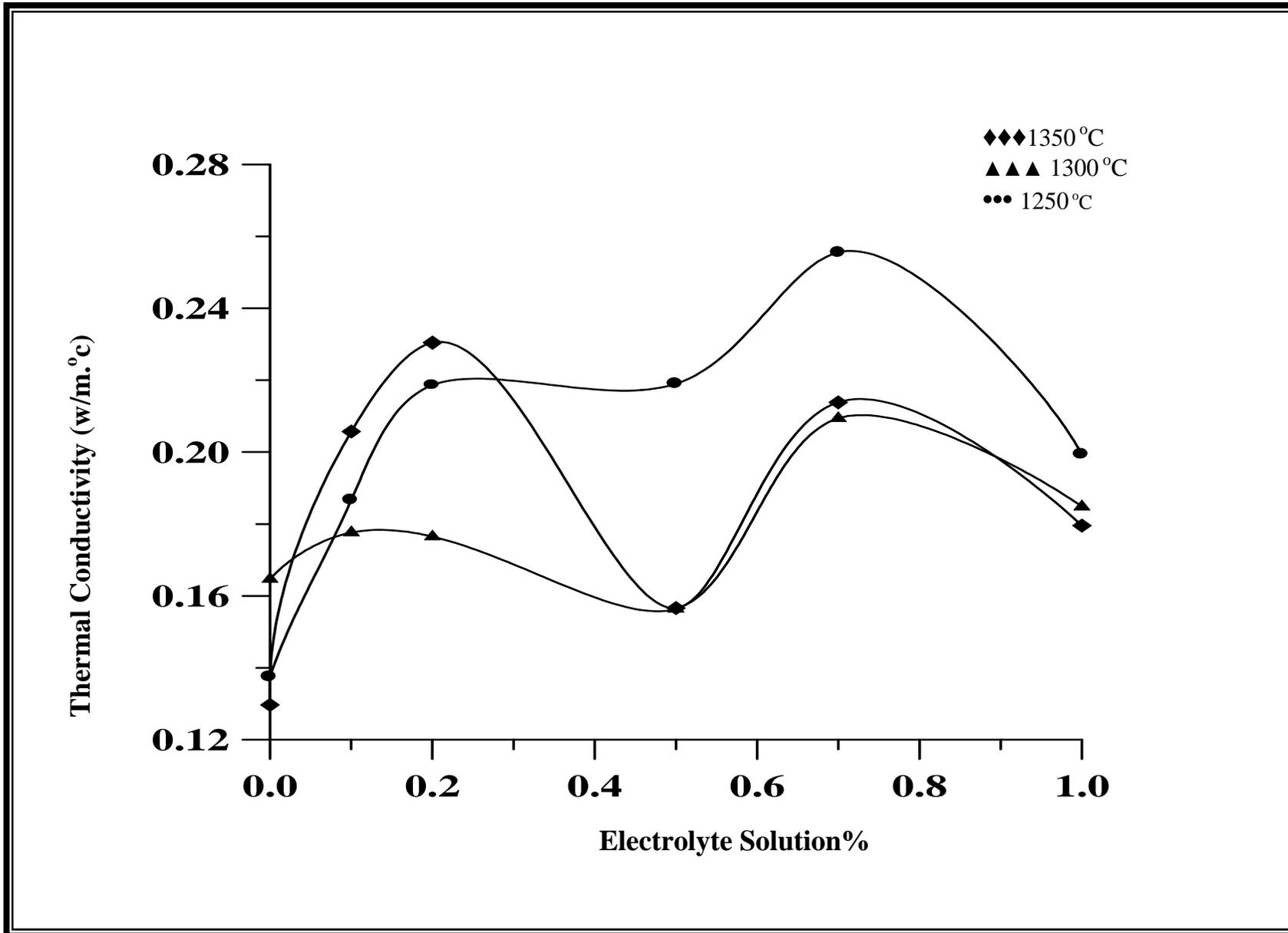
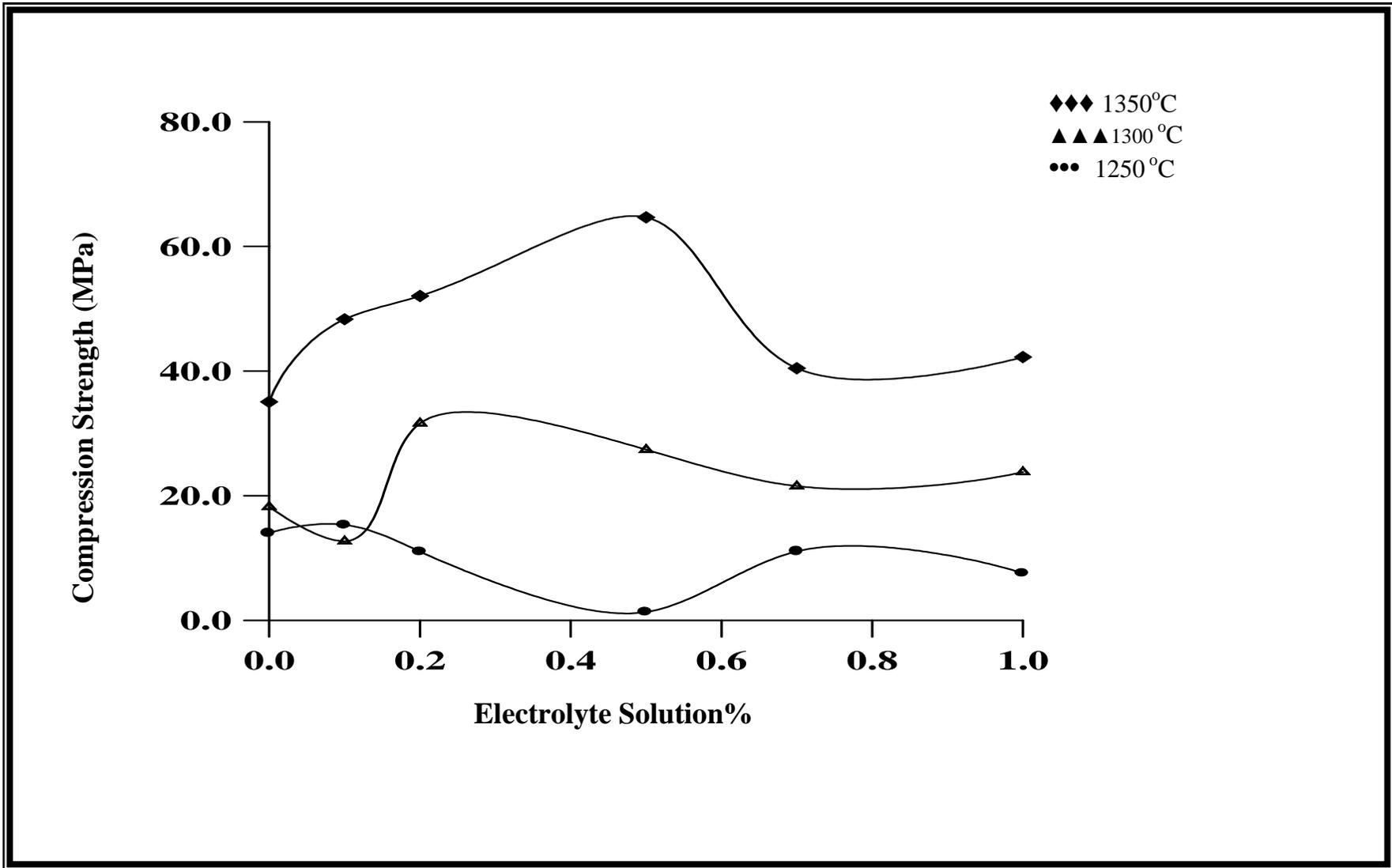


Figure (4-7). The thermal conductivity of the samples with different concentration of electrolyte solution as a function of sintering temperatures.

When each particle is completely surrounded by a film of water of the required thickness, the addition of further water will effect a reduction in the strength of the fired articles due to mainly to the increase in porosity which results. So the requirement is to add bonding agent, which may seriously affect the strength of the penetrated material. This bonding agent is electrolytes, when added to clay in form of pastes and slips, considerably increase the strength.

The mechanism by which the strength of materials is increased by the addition of electrolyte is probably intimately linked to the deflocculating effects. The particles are more readily dispersed in water when electrolyte are present and a better distribution of plastic material is there by achieved. The limit of adding bounding agent is controlled by so many parameters; these parameters are effect of shaping, drying, firing process, effect of duration of firing, and effect of cooling.

In this study these parameters are controlled with process defined in practical part, so practically found 0.5% specified electrolyte solution given a highly compression strength values for porcelain body firing at 1350°C. From fig. (4–8) one can find the effect of firing temperature on compression strength, in wide range of electrolyte solution as the firing temperature increase, the compression strength increase because of liquid phase effect.



Figure(4-8). The Compressive Strength for different concentration of electrolyte solution as a function of sintering temperatures

### **4.3 Conclusion:**

1– The dielectrics, physical and mechanical properties measuring without adding 2% ZnO given result lower than the group using 2% ZnO this indicator that adding ZnO mineralized improved these properties. The best percentage of electrolyte solution which used to produce a good electrical insulator, 0.2% at sintering temperature (1350 °C).

2– Physical and mechanical properties affected by adding electrolyte solution in concentration(1%, 0.7%, 0.5%, 0.2% and 0.1%) and the best result obtain at 0.5%.

3 – The values of Physical and mechanical properties at 0.2%concentration of electrolyte solution consider acceptable value, therefore 0.2%concentration of electrolyte solution is optimized value in electrical insulator from Iraqi clays.

### **4.4 Future Work:**

1– Using other values of ZnO percentage and study the effect on the improving of the dielectric properties of porcelain insulator.

2–Studying the effect of using anther minlaizer material such CaO, MgO and TiO for samples have good dielectric properties.

3– Perform mechanical measurements on the dielectric samples such as bending.

4– Dielectric properties such as Dc–resistivity for insulator samples.

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## الخلاصة

يهدف البحث الى دراسته تأثير المحلول الالكتروليتي و نسب تركيزه على الخواص الكهربائية والفيزيائية لجسم البورسليني الذي تم تحضيره بأستخدام مواد محليه عراقية. تم تشكيل الجسم بور سليني من كاؤولين دويخله، رمل زجاج أرضمه، فلدسبار وبنسب وزنيه (45%، 30%، 25%) على التوالي. ولقد أجريت معاملة كيميائية لكل من الكاؤولين و السليكا بهدف تنقيتها من الشوائب التي يمكن أن تذوب بحامض الهيدروكلوريك حيث تمت دراسة التغيرات الحاصلة من خلال تحاليل IR .

قسمت الدراسة إلى سبعة مجاميع محضره من هذه المواد طبقا لنسب المضافة من المحلول الالكتروليتي المحضر من  $(\text{NaCO}_3 + \text{NaSiO}_3)$  و بنسبه (2:1) أن نسب إضافات المحلول الالكتروليتي هي (1%، 0.7%، 0.5%، 0.2%، 0.1%) حيث ثبت نوع و نسبه الماده المعدنه في تشكيل الجسم البورسليني و هي أو أكسيد الزنك  $\text{ZnO}$  وبنسبه (2%). تم تشكيل مكبوسات من هذه الخلطات وبعد التجفيف تم حرقها لدرجات حرارة مختلفة ( $1350^\circ\text{C}$ ) (1250, 1300)

أجريت القياسات العزليه الكهربائيه (ثابت العزل و معامل الفقدان العزلي) وكذلك التوصليه الحرارية و قابليه الانضغاط .

أن النتائج المتحققة لنماذج المحروقه بدرجه  $1350^\circ\text{C}$  أ بدت نمط من ثبوت قيم العزليه الكهربائيه عند حدود معينه من تراكيز المحلول الالكتروليتي المضاف لمكونات الجسم البورسليني ضمن مدى (0.1 – 0.2) ، في حين هنالك تغير في قيم التوصليه الحراريه ضمن مدى (0.2 – 0.5%) من تركيز المحلول الالكتروليتي. وأن قابليه الانضغاط تكون عاليه عند حدود (0.5%) من تركيز المحلول الالكتروليتي لنفس المكونات، وبالتالي يتطلب اعتماد مفاضلة في الخواص وحدودها لاختيار افضل تركيز المحلول الالكتروليتي بما يوفر توافق نتائج الخواص العملية مع الخواص الصناعيه القياسيه المتحققة على منتجات محضره من مواد بلدان أخرى ومعتمده لديهم ، لقد تم اعتبار أن افضل تركيز يتوافق مع الخواص العزليه الكهربائيه وكذلك التوصليه الحرارية والميكانيكية هو (0.2%) كما اشاره إليه النتائج.



جمهورية العراق  
وزارة التعليم العالي والبحث العلمي  
جامعة النهرين/كلية العلوم  
قسم الفيزياء

# دراسة تأثير المحاليل الالكتروليتيه على خواص العزليه الكهربائية للبورسلين المشكل من مواد محليه

رسالة

مقدمة إلى كلية العلوم - جامعة النهرين

وهي جزء من متطلبات نيل درجة الماجستير في الفيزياء

من قبل

عتاب فاضل حسين

بكالوريوس ٢٠٠٢ (جامعة النهرين)

بأشراف

د.أزهار جواد داوود

د. فاضل عبد رسن

١٤٢٦هـ  
٢٠٠٥م

ربيع الأول  
نيسان

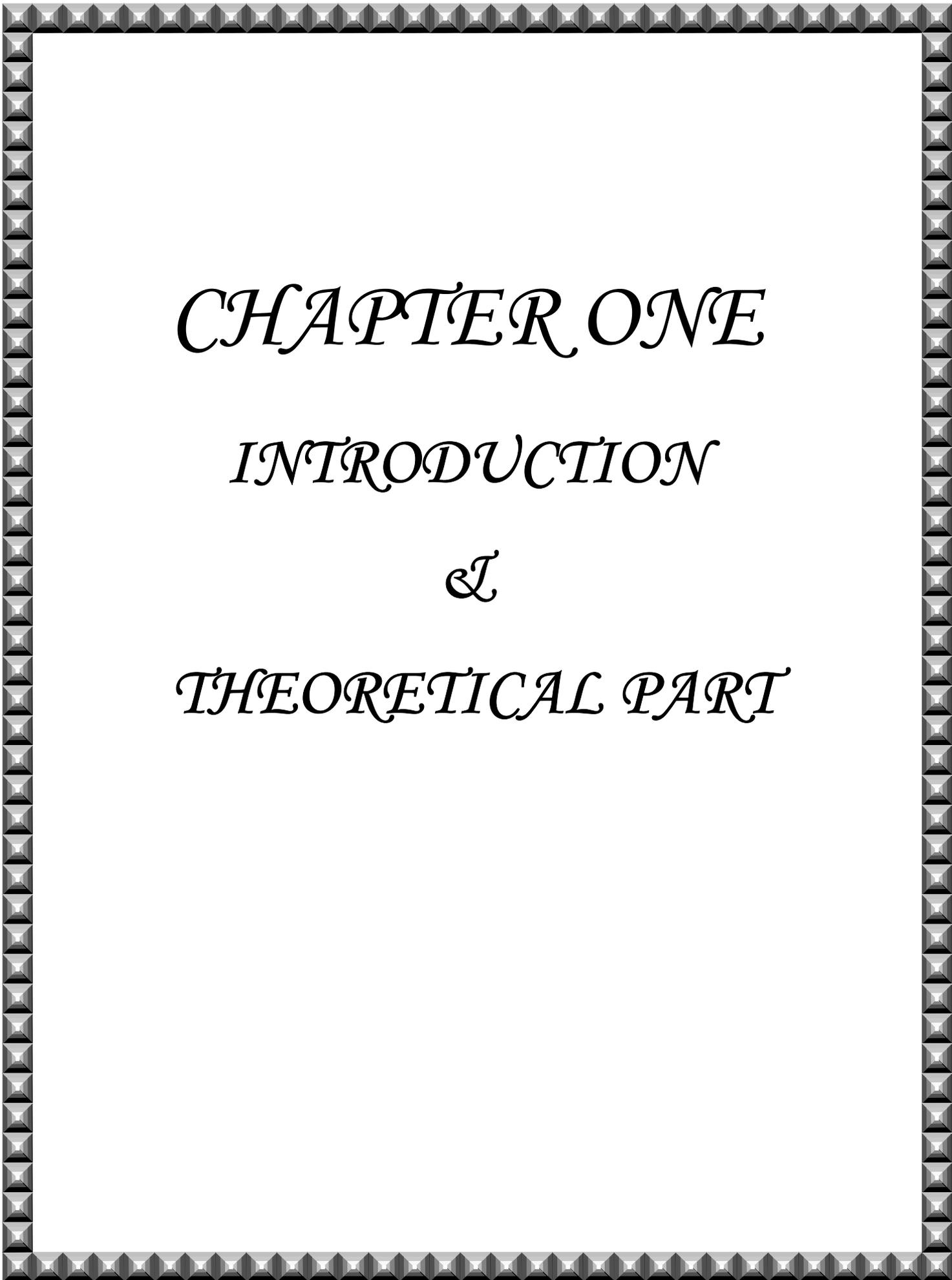
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وَعَلَى وَالِدِي وَأَنْ أَعْمَلَ صَالِحاً تَرْضَاهُ وَأُدْخِلْنِي

بِرَحْمَتِكَ فِي عِبَادِكَ الصَّالِحِينَ

صَدَقَ اللَّهُ الْعَظِيمُ

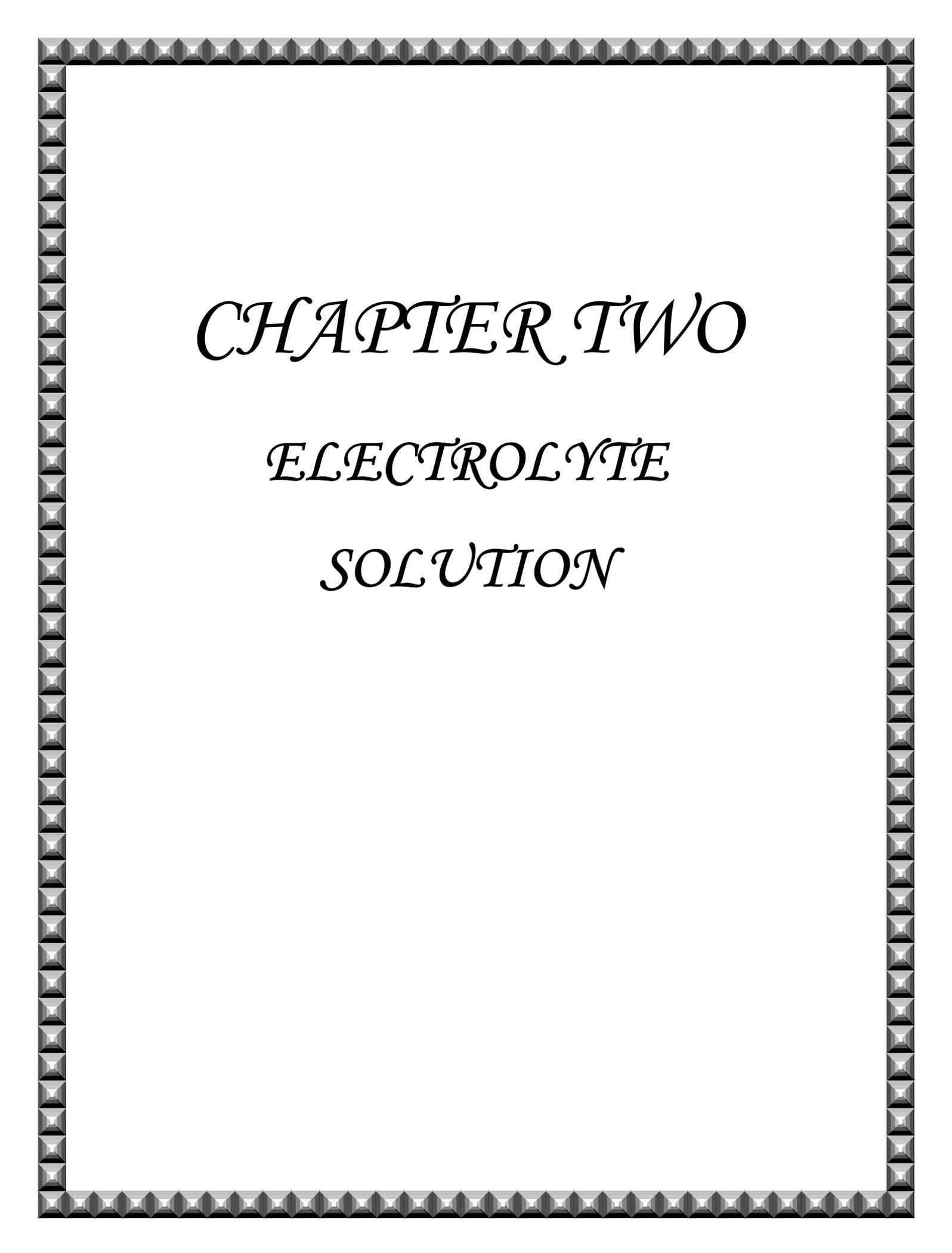


*CHAPTER ONE*

*INTRODUCTION*

*&*

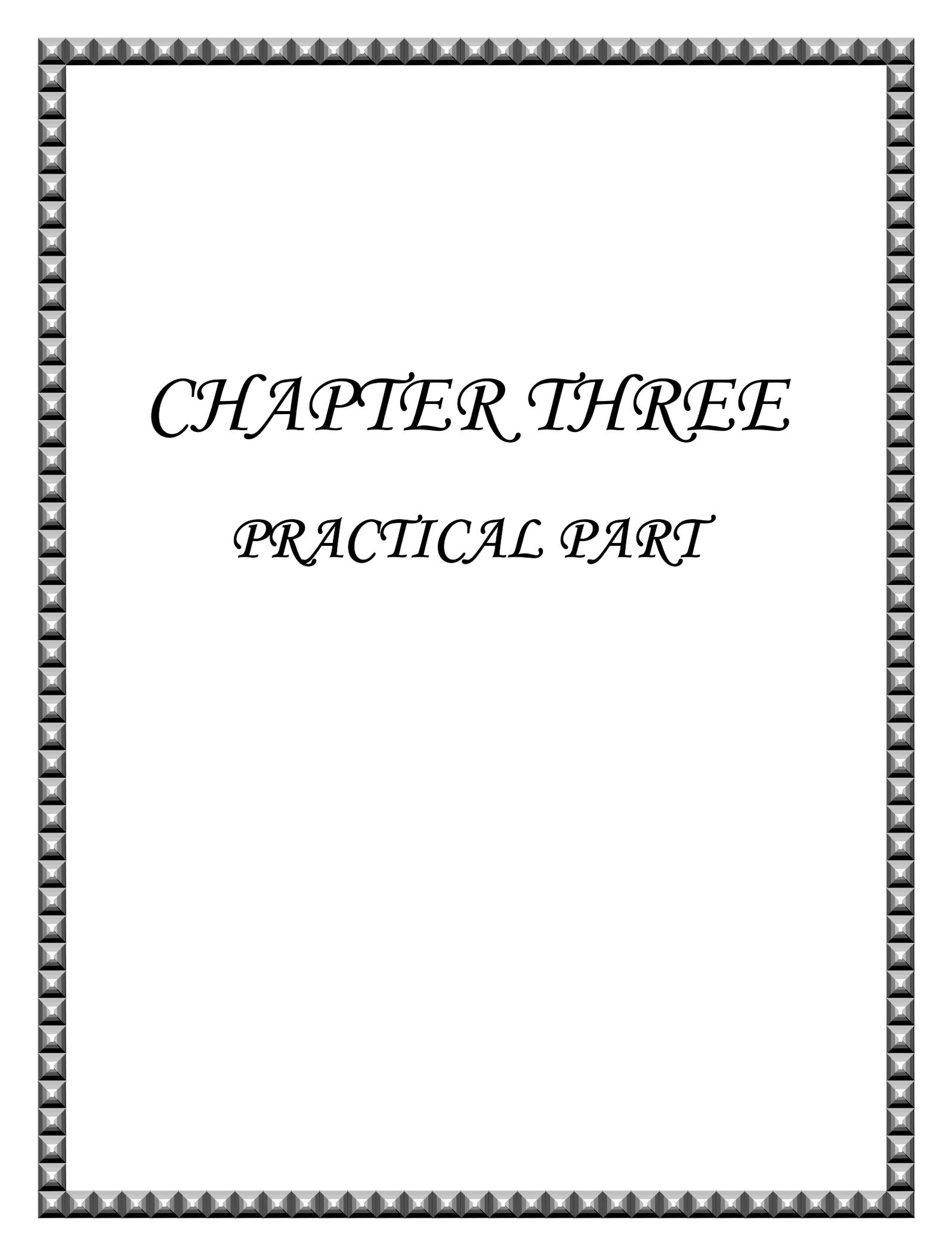
*THEORETICAL PART*



*CHAPTER TWO*

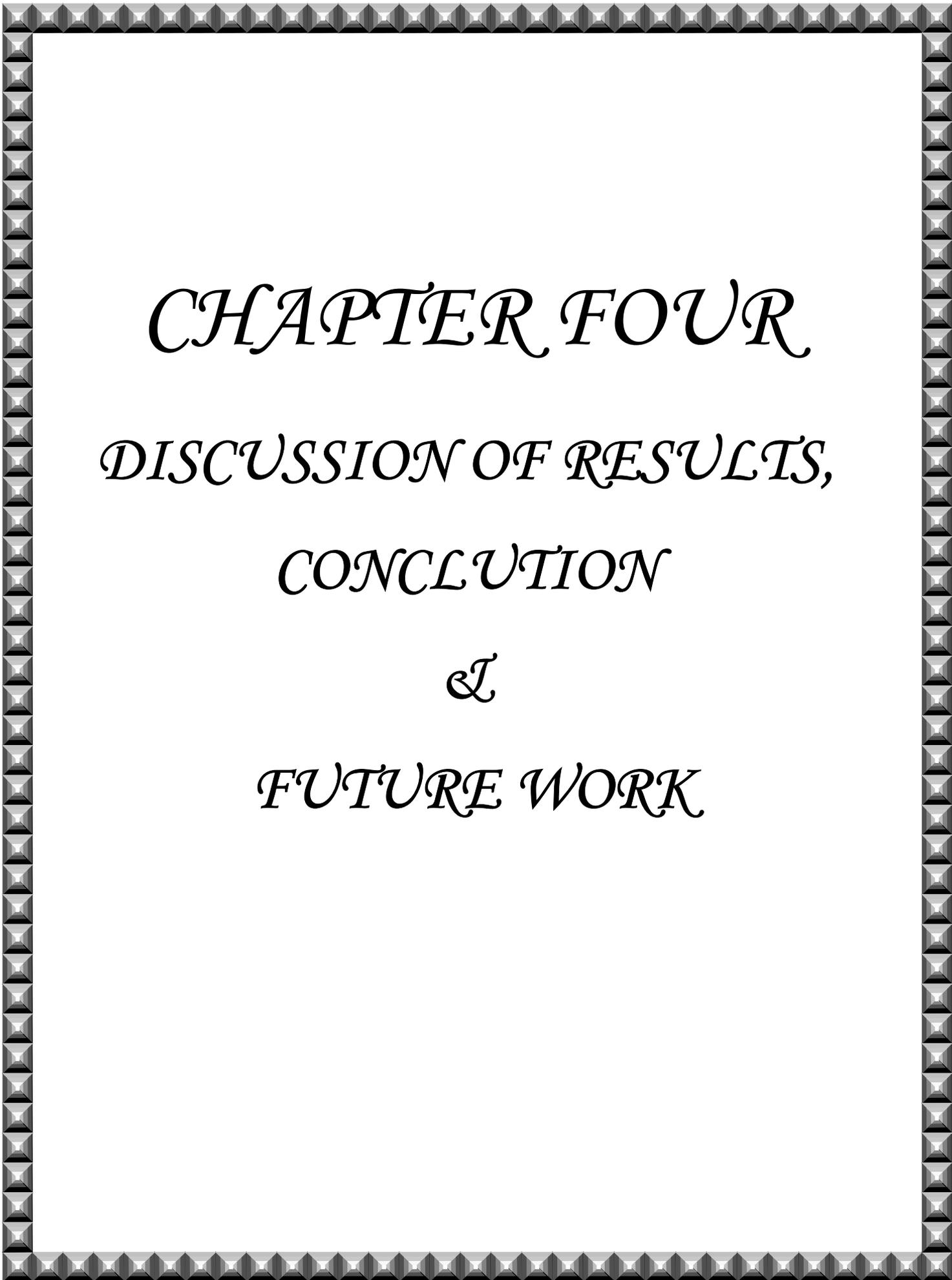
*ELECTROLYTE*

*SOLUTION*

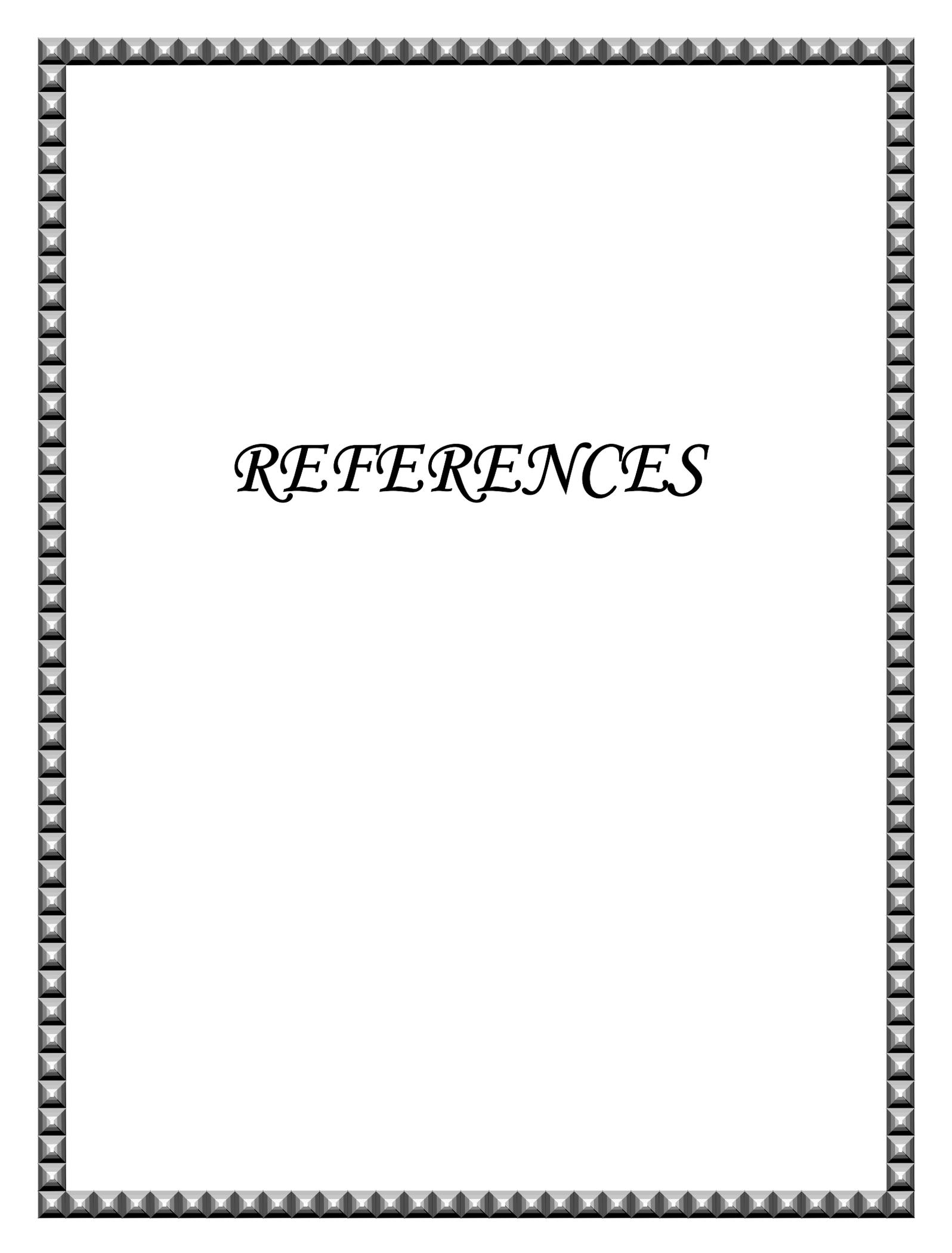


*CHAPTER THREE*

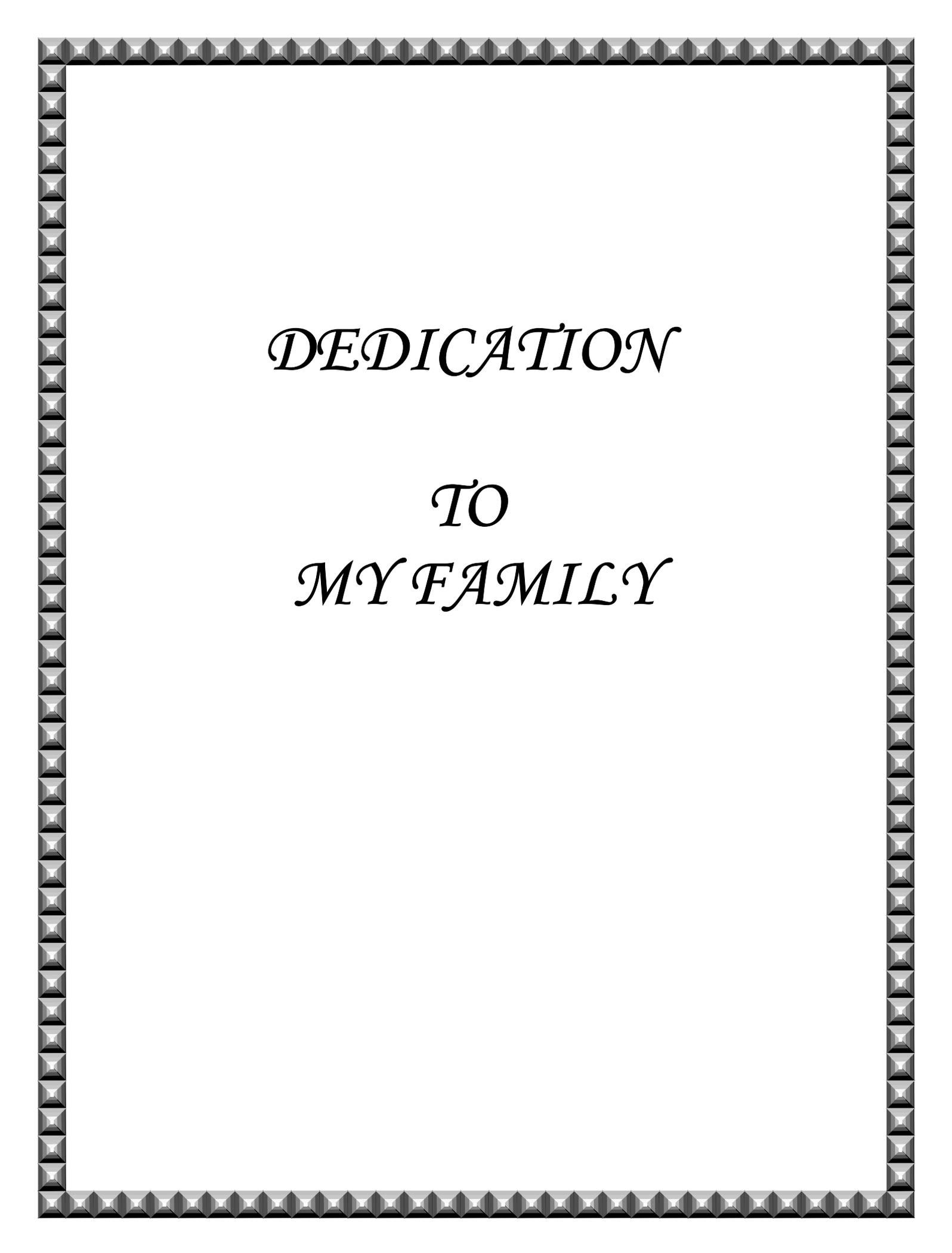
*PRACTICAL PART*



*CHAPTER FOUR*  
*DISCUSSION OF RESULTS,*  
*CONCLUSION*  
*&*  
*FUTURE WORK*



*REFERENCES*



*DEDICATION*

*TO*  
*MY FAMILY*