Kurdistan Regional Government Ministry Higher Education & Scientific Research Salahaddin University College Of Education Department Of Chemistry



DEVELOPMENT OF GLASS CERAMICS FROM KAOLIN, SODALIME AND BROSILICATE WASTE

A Project Submitted To The Council Of The College Of Education At Salahaddin University In Partial Fulfillment Of The Requirements For The Degree Of B.Sc. At Chemistry Department.

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Supervisor recommendation

I am the student's supervisor, -----student full name----Sherwan Rahman Sofi---. I support that the student has completed all the requirements for submitting the research drawn entitled -------title of project-- DEVELOPMENT OF GLASS CERAMICS FROM KAOLIN, SODALIME AND BROSILICATE WASTE-- according to the numbered administrative order 3/1/5/1972 on 9th oct. 2022 in accordance with the instructions of Salahaddin university quality assurance and it is ready for discussion.

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Dedication

I'd want to give credit to my parents and grandparents for giving me the opportunity to have a good education from a young age and instilling in me a love of learning. I would like to convey my thanks to all my professors who have guided me and encouraged me during my academic study.

Acknowledgment

(In the name of Allah, most Kindness and most Merciful)

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Abstract

Recycling and reprocessing of hazardous and non-hazardous waste materials is essential for environmental protection and economic benefits. Borosilicate glass has a wide range of applications due to its chemical durability and low thermal expansion. Borate mineral wastes obtained through boron mines were used to study waste mixtures. Thermal decomposition of kaolinite 2SiO2Al2O3H2O produces the aluminosilicate named "mullet" with a segregation of amorphous silica which crystallizes as cristobalite by heating. Soda-lime glass is composed of SiO4 tetrahedra connected to four oxygen atoms and each oxygen atom shared by two silicon atoms. Network modifiers such as monovalent Na2O ions disrupt the continuity of the network, making it more difficult for the atoms to arrange themselves for crystallization to occur.

Glass-ceramic production involves the movement of atoms/molecules from the glass to the crystal, resulting in unique properties such as chemical and mechanical.

Key words: Recycling, Atoms, Thermal, Soda-lime.

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CHAPTER ONE

1.Introduction

Industrial processes, manufacturing, and human activities are always accompanied by hazardous and non-hazardous waste materials. Thus, recycling and reprocess of such wastes is essential for environmental protection and maximizing economic benefits. Recycling is the selection, classification (Pelino, 2000), and reemployment of waste as a raw material to produce the same, or very similar product, to the parent material Glass waste is abundant particularly in urban areas due to intensive usage of glass in many daily life activities.

Borosilicate glass, in particular (Appendino, Ferraris, Matekovits, & Salvo, 2004), has a wide range of applications due to its chemical durability and low thermal expansion, that vary between domestic purposes (Pyrex cooking wares) (Stoch, 2004) scientific purposes (laboratory glassware), medical purposes (glass ampoule), and industrial applications involving reshaping of borosilicate glass tubes . Such excessive usage of borosilicate glass produces a large amount of waste (Plodinec, 2000), Moreover, glass waste should get good attention for environmental protection, especially it takes millions for year for glass to degrade in nature. So, the recycling of borosilicate glass represents a great issue that should be studied and could have great economic and environmental impacts. Reduction of energy consumption is an add-up advantage for using glass in the ceramic industry which will lead to reducing the overall cost of ceramic materials.

Glass-ceramic composites are materials that are originally processed as glasses and converted to glass-ceramic composite to improve the properties of ceramics (e.g., chemical durability). Borosilicate glass typically is composed of 70–80 wt% silica, 7–13 wt% of boron oxide, and 4–8 wt% sodium oxide or potassium oxide, and 2–8 wt% of aluminum oxide. Boron trioxide in borosilicate glass allows for a very low thermal expansion coefficient. In contrast, soda-lime glass thermal expansion coefficient is more than double that of borosilicate glass and can be easily cracked under temperature versions (Porter & Emery, 1938), Therefore, recycling of borosilicate glass was our choice rather than soda-lime glass for the production of low thermal expansion glass-ceramic. Glass ceramic could be also used in metallurgy and steel production, glass manufacturing, solid-oxide fuel cells (SOFCs), nuclear reactors, aerospace, automobile components, protective ceramic coatings, insulator, and industrial tooling (Bernardo, Bonomo, & Dattoli, 2010), The resistance of borosilicate glass to chemicals is so high that it is used to store nuclear waste (Lima & Monteiro, 2001).

The boron in the borosilicate makes it less soluble, preventing the leaching of any harmful materials from glass to surrounding environment. Such superior property makes borosilicate glass is good choice to produce chemically durable glass-ceramic composite. Highly chemical durable ceramic and glass-ceramic composites find applications in laboratory accessories such as mortar, ceramic parts of desiccator, and ceramic proppants (Wendler et al., 2017). In this work, borosilicate glass waste was recycled through the preparation of borosilicate glass-ceramic composite (BSc). Borosilicate glass waste was thermally treated by the sintering process of borosilicate glass and kaolin. The prepared BScs has the advantages of production at relatively low temperature, low thermal expansion coefficient, and high chemical durability (see belowIn this work, borosilicate glass waste was thermally treated by the sintering composite (BsGC). Borosilicate glass waste was thermally treated by the sinter glass-ceramic composite (BsGC). Borosilicate glass waste was thermally treated by the sinter glass-ceramic composite (BsGC). Borosilicate glass waste was thermally treated by the sintering process of borosilicate glass and kaolin.

The prepared BScs has the advantages of production at relatively low temperature, low thermal expansion coefficient, and high chemical durability, Glass-ceramic materials feature a great potential for environmental clean-up, since they may combine waste stabilization and valorization. In present research, considering the main aim of the thesis, as environment friendly waste utilization, energy consumption was also noticed, in order support environment respecting production techniques. For this concern a controlled fast heating rate, ranging between 40°C min-1 to 2 °C min-1 was applied to studied waste mixtures. The borate mineral wastes obtained through boron mines and enrichment plants were selected to be the constant starting material in all performed studies. Borate mineral wastes contain high amount of B2O3, a very well-known glass forming oxide with a large scale of application areas, also providing lower sintering temperatures.

The utilization of borate mineral wastes with glass-ceramic technology was first time studied and primarily not investigated combinations of wastes were incorporated into the research. These wastes consist of; soda lime silica glass, meat bone and meal ash and fly ash. In order to investigate possible and relevant application areas in ceramics, kaolin clay, an essential raw material for ceramic industry was also employed in some studied compositions. Different proportions prepared according to the objected glass-ceramic ternary systems such as; B2O3–P2O5–SiO2 (borophospho-silicate) and SiO2-Al2O3-CaO (wollastonite). In order to determine cytotoxicity, cell toxicity analyses applied to some of the studied products, in order to see the effect of boron (a regular daily intake element) in glass structure.

CHAPTER TWO: LITERATURE RIVEW

2. Determination off chemical oxide composition of waste kaolin

is а dioctahedral phyllosilicate with Kaolinite а theoretical formula Al2O3·2SiO2·2H2O or Al2Si2O5 (OH) 4 with a 1:1 layered structure of [(Si2O5)] 2- and [Al2(OH)4] 2+ molecular sheets and composition of 39.5 wt. % Al2O3, 46.5 wt. % SiO2 and 14 wt. % H2O (Francis, Rawlings, & Boccaccini, 2002). According to Murray (Pask & Tomsia, 1991). kaolinite can be formed by hydrothermal alteration or weathering reactions or by a combination of both processes. However, Galan and Ferrell (Sanz, Madani, Serratosa, Moya, & Aza, 1988). indicated that kaolinite can be found also by digenetic processes. Clay minerals, as kaolinite, are formed by decomposition of Feld spathic rock via geological processes (Carty & Senapati, 1998). On the other hand, feldspars are the most widespread mineral group in the world forming around 60% of the earth's crust . (Murray, 1988). suggested a typical reaction sequence, using potassium feldspar (KAlSi3O8), as follows:

$\begin{array}{rrrr} 2\text{KALSI3O8} & + & 3\text{H2O} & \rightarrow \text{Al2Si2O5} & (\text{OH}) & 4 + & 4\text{SiO2} & + \\ 2\text{KOH} & & & (\text{REACTION 1}) \end{array}$

It has been mentioned that if potassium ions are not properly removed (following the weathering process), illicit clays (2:1 layered structure silicates) are formed instead of kaolinite Kaolin ores, with a relative high proportion of kaolinite, are a very interesting raw materials, with important and wide applications due to the properties of kaolinite layer silicate: low surface area (compared to other clay minerals, such as spectates), layered appearance, white or almost white color, chemically inert in a wide range of pH (4–9), with low heat and electricity conductivity, refractoriness (high-temperature resistance of ~1550 °C) and non-abrasive [Galan et all.,2013:83-126]. Consequently, powdered and processed raw kaolin materials with a relatively high-proportion of kaolinite can be technologically applied in several industries, such as porcelain manufacture, ceramics, refractories, paper, paints, composite materials, polymers, and many more [Deer et all.,1992]. On the other hand, thermal decomposition of kaolinite 2SiO2·Al2O3·H2O produces the aluminosilicate named "mullet" with composition 3Al2O3·2SiO2 in high relative proportion, with a segregation of amorphous silica which crystallizes as cristobalite by heating (Dondi, 2018). The summary reaction is as follows

3 2H2O 3A12O3·2SiO2 4SiO2 6H2O

(REACTION 2)

2.1. Formulation of soda lime glass waste:

Soda-lime glass, also called soda-lime-silica glass, is the most prevalent type of glass. It is composed of SiO4 tetrahedra connected at the oxygen atoms. The chemical ordering is very strong; each silicon atom is connected to four oxygen atoms and each oxygen atom is shared by two silicon atoms, as shown in Fig. 1.

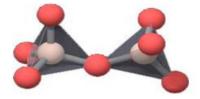


Fig.1 The Si–O–Si bond

In soda-silica glass, the continuity of the network is disrupted by the addition of network modifiers, such as monovalent Na2O ions (Murray, 1988). These network modifiers make the network more sophisticated so that when the components are melted together during the cooling process, it is more difficult for the atoms to arrange themselves in suitable configurations for crystallization to occur (Chen, Lan, & Tuan, 2000). Fig. 2 shows adjacent SiO4 tetrahedral-type unit cells forming part of a continuous random network.

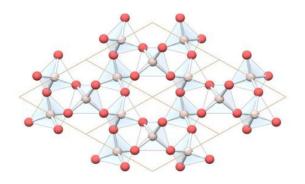


Fig. 2 Adjacent SiO4 tetrahedral-type unit cells

Soda lime glasses have a typical composition (wt.%) and small amounts of other oxides. These glasses are employed for high-volume products such as windows, bottles and jars. The added metal oxides act as network modifiers in the structure of Fig. . Thus, when soda (NaO2) is added to silica glass, each Na+ ion becomes attached to an oxygen ion of a tetrahedron thereby reducing the cross-linking as indicated in Fig. . The effect of soda addition is thus to replace some of the covalent bonds between the tetrahedra with (non-directional) ionic bonds of lower energy. This reduces the viscosity of the melt, so that soda glass is easily worked at 700 °C, whereas pure silica softens at about 1200 °C. By the same token, this alloying of the glass to make it more workable reduces its strength at high-temperature, so that silica glass must be used in applications requiring high-temperature strength – such as the envelopes of quartz halogen lamps.

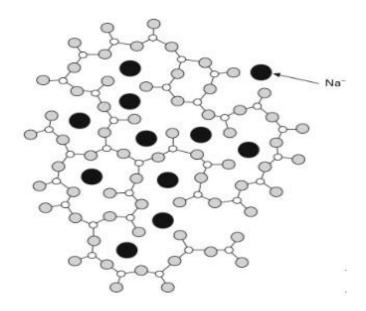


Fig.3 Sketch of the structure of soda lime glass.

Soda lime glasses are also sensitive to changes in temperature, and, because of their large coefficient of thermal expansion ($\sim 8 \times 10-6$ K–1), they may develop high thermal stresses that can induce cracking. The second important family of glasses were developed to overcome this problem.

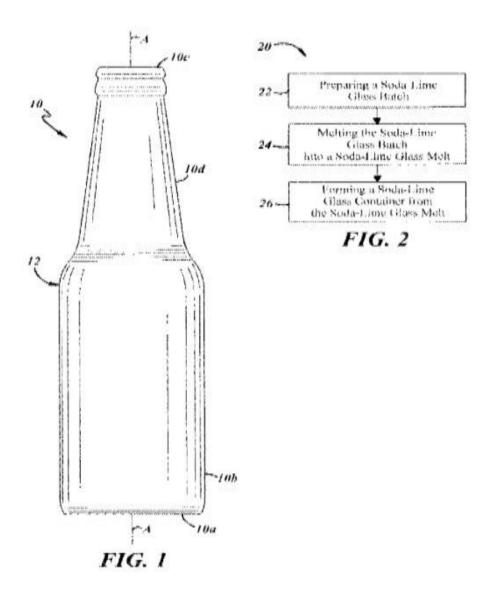
2.2. Determination the centering temperature on the properties of the glass ceramic:

The soda-lime glass batch may be melted (step 24) in one or more furnaces to produce the soda-lime glass melt. The temperature of the fumace(s) is set to ensure proper melting of the glass batch according to known practices. For example, to produce the soda-lime glass melt, the glass batch may melted in the fumace(s) at a temperature about 50°C lower than the temperature of a melt of a typical glass batch (with 30-40 wt.% cullet) which is usually between about 1400°C and about 1500°C, at a typical residence time of about two to four hours. After achieving its melt form, the soda-lime glass melt may flow from the fumace(s) into a refiner, where it is conditioned, and then to one or more (Castelein, Soulestin, Bonnet, & Blanchart, 2001).

The soda-lime glass container 10 (step 26) may then beformed from the soda-lime glass melt by a glass-blowing procedure. A feeder located at a downstream end of the oneor more forehearths, for example, may measure and deliver agob of the soda-lime glass melt to a glass-forming machine. The gob may then be formed into the soda-lime glass wall 12 at an individual section machine by a press-and-blowprocess, a blow-and-blow process, or any other suitable process. Once formed, the soda-lime glass wall 12 is initially cooled to preserve its desired shape, and then annealed inone or more an annealing lehrs. The soda-lime glass wall 12 may be annealed at a hot-end portion of the annealing lehr(s) at a temperature between about 550°C and about 600°C forabout 30 minutes to about 90 minutes, and then graduallycooled at a cold-end portion to between about 65°C and about 130°C. Any of a variety of hot-end, cold-end, antireflective, and/or glass strengthening coatings may be plied to the exterior of the soda-lime glass wall 12 anytime after being formed (Yamuna, Devanarayanan, & Lalithambika, 2002).

A soda-lime glass batch was prepared that included 100 wt.% cullet as the glass forming materials. The cullet used was supplied as a mixture of several different types of glass. Specifically, the supplied cullet included the following mixture: 40-48 wt.% green glass, 42-50 wt.% flint glass, 6-14 wt.% amber glass, and 0-2 wt.% blue glass. The supplied cullet also included less than 250 g/ton of opal glass, less than 1000 g/ton of organics, less than 100 g/ton of plastics, less than 25 g/ton of ceramics, less than 5 g/ton of magneticmetals, and less than 5 g/ton of non-magnetic metals (Chen et al., 2000). There thus has been disclosed a process for making soda-lime glass that fully achieves all of the objects and aims previously set forth. The disclosure has been presented in conjunction with presently preferred

embodiments, and alternatives and modifications have been discussed. Other alternatives and modifications readily will suggest themselves to persons of ordinary skill in the art in view of the foregoing description.





Glass-ceramics are polycrystalline materials obtained through controlled crystallization (devitrification) of selected/suitable glass compositions. Due to characteristics of glasses, the decided composition and its heating treatmentshave to be well studied. Today, not only glass compositions but also different heat treatments are still discussed in respect of producing glass ceramics1. There are limited glass composing precursors for glassceramics production. Compared to stable and difficult crystallizing parent glasses, some glasses crystallize fast (spontaneous crystallization) and uncontrollably, resulting in undesirable microstructure making impossible to employ a preferred structure. Glass-ceramics have a glass (amorphous) phase and typically one or twocrystalline phases usually in the range from 30 vol% to 95 vol %. Glass- ceramics share the unique characteristics and advantages of glasses as well as ceramics, exhibiting significant advantages compared to conventional glass orceramic materials. Glass-ceramics technology provides materials having high mechanical strength and toughness, low (even negative) thermal expansion, high thermal strength. A widely known example for heat resistant glass- ceramic is Li2O-Al2O3-SiO2 system.

2.3. Glass Ceramics from Wastes

Thermal treatments of problematic wastes by glass-ceramic technology has been welldocumented regarding its effectiveness in immobilising the toxic contents in their glass matrices (Strnad, 1986). These kinds of heat treatment technologieshave been widely used for treatment of several wastes usually processed to form glass-ceramic products [Rawlings and Boccaccini,2006:733-761]. These wastes, coming from numerous sources, can be also considered raw materials and comprise, as main examples (Holland & Beall, 2002)

- Slag from metallurgical processes (iron and steel production)
- Coal Ash from Power Stations
- Residues from Urban Incinerators
- Slag from Gasification Processes38
- Electric Arc Furnace Dust (Steel Fly Ash)
- Cement Dust
- Ore-Refining Quartz-Feldspar Waste
- Fluorescent Glass Waste
- Sewage Sludge
- Anodising Plant Industrial Waste
- Zinc-Hydrometallurgy Wastes
- Clay-Refining Waste (Kira)[Manfredini et all.,1997]

2.3.1. Crystallization (devitrification)

The transformation of glass to form glass-ceramic is called crystallization, also known as devitrification. This transformation consists of two parts, called nucleation and growth. Nucleation is the key part in controlling the crystallisation where crystalline glass phases occur. [Davies et all.,1970:37-348] There are two types of nucleation: the first one is volume nucleation, which is commonly used technique for both homogenous and heterogeneous nucleation. The second type is surface nucleation by which is more difficult to control the crystallization. The nucleation (Berezhnoi, 2012). process mostly ends with an undesired microstructure setting the characteristics of the glass ceramic. So, it must be never forget that largeoriented structure exhibits better mechanical properties. In some parent glass compositions, nucleating agents are needed; these agents can be either metallic or non-metallic due to envisaged characteristics of the glass-ceramic. Nucleation through nucleating agent is called internal nucleation or bulk (Ferreira, Ribeiro, & Ottosen, 2003).

2.3.2. Nucleation

The growth part takes place after obtaining a nucleus matching desired characteristics. The transformation in growth part is movement of atoms/molecules from the glass, across the glass-crystal (Erol, Demirler, Küçükbayrak, Ersoy-Meriçboyu, & Öveçoğlu, 2003). interface and into the crystal. The growth part takes place after obtaining a nucleus matching desired characteristics. The transformation in growth part is movement of atoms/molecules from the glass (Francis et al., 2002), across the glass-crystal interface and into thecrystal2, a temperature depending treatment to grow the grains54. A typical processing cycle for Li2O-Al2O3-SiO2.

2.4. Glass-Ceramic Production Routes

An increasing amount of investigation on glass-ceramic production has been made in recent years. These processing routes differ from each other in order to obtain unique properties such as expected microstructural or mechanical properties. The glass-ceramic production techniques can be listed as;

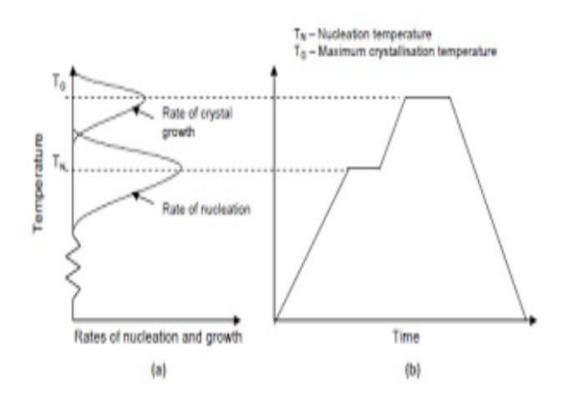


Fig 5 (a) & Fig.5 (b)

2.4.1. Conventional Method (two-stage)

Devitrification of glass by two-stage heat treatment is the conventional method for producing a glass-ceramic (Figure 5 (b). The first stage is a low temperature heat treatment at a temperature that gives a high nucleation rate (around TN in Figure 5a) thus forming a high density of nuclei throughout the interior of the glass. A high density of nuclei is important as it leads to a desirable microstructure consisting of a large number of small crystals. The second stage is a higher temperature heat treatment at around temperature TG [Kim and Kim,2004:2825-2833], to produce growth of the nuclei at a reasonable rate.

2.4.2. Modified Conventional Method (single-stage)

The double-stage heat treatment of glass in the conventional method is due to the limited overlap between the nucleation and growth rate curves (Figure 5 (a). If there is extensive overlap of the rate curves then nucleation and growth can take place during a single-stage heat treatment at temperature TNG as indicated in Figure 5. The rate curves, particularly the nucleation rate curve, is sensitive to composition, hence by optimizing the glass

composition and the included nucleating agents it is, in some cases, possible to obtain the necessary overlap. The pioneer glass-ceramic system utilized thismethod is known as —Silceraml (Pask & Tomsia, 1991). as will be explained in the next section.

2.4.3. PETRURGIC METHOD

It was found with —Silceraml that it made little difference whether the glass was heated up to TNG from room temperature or the molten glass was cooled to TNG (Ferreira et al., 2003). This directed scientist to produce certain glass-ceramics by a controlled, very slow cooling of the parent glass from the molten state directly without a hold at an intermediate temperature. With this method, referred to in the recent literature as the Petrurgic (Hing, Sinha, & Ling, 1997). both nucleation and crystal growth can take place during the cooling. Both the modified conventional (single-stage) and the Petrurgic methods are more economical that the conventional method (two-stage).

2.4.4. Powder Methods

High temperature heat treatment (sintering) of cold-compacted powder is the common method for the fabrication of ceramics and it has been likewise used for glass-ceramic manufacture (Hing et al., 1997). As there are limitations on the size and shape of components that may be cold compacted, and also a cost in producing a powder, this method is only used if an obvious benefit is identified. In most cases there is little advantage in compacting and sintering a glass-ceramic powder because a high sintering temperature is required and the resulted glass-ceramics do not have significant better properties than those manufacturing by other techniques. It is more attractive to sinter a parent glass powder, which sinters by a viscous flow mechanism at lower temperatures .It is important to consider the rates of viscous flow sintering and crystallization and the interaction of these processes. If crystallization is too rapid the resulting high degree of crystallinity will hinder the low temperature sintering leading to an unacceptable amount of porosity (Lambrinou, Van der Biest, Boccaccini, & Taplin, 1996).

2.4.5. Sol-Gel Method

glasses produced from the molten state have been considered but in the last decades there has been considerable interest in using sol-gel and colloidal techniques to obtain the precursor glass in either powder or bulk form (Lambrinou et al., 1996) Glass-ceramics produced by all the previous routes could be produced by sol-gel technique except that; they can't be manufactured from waste materials.

2.5. Determination the physical, chemical and mechanical properties of the glass:

2.5.1. CHEMICAL PROPERTIES

All soda lime type glasses and some borosilicate glasses contain sodium or alkali metal ions. Prolonged exposure to liquids or vapor, such as water, will cause the sodium/alkali ions to migrate to the surface of the glass called sodium or alkali leaching. This can cause cloudiness or haze on the surface of the glass. Porous coatings may also incur this phenomenon, causing a disruption of the bond between the coating and the glass surface. In high humidity or critical surface applications, this must be considered when specifying the material. Placing a "barrier" coating, such as silicon dioxide, on the glass will limit the amount of reaction. The acid resistance and alkali resistance measure the time it takes to remove a layer of specified thickness for each test. (Scherer, 1977)

2.5.2. Mechanical properties

The mechanical properties of glass determine the amount of stress a glass can withstand. Stress is defined as the perpendicular force per unit area applied to an object, in a way that compresses (compressive stress) or stretches (tensile stress) the object. Strength of the ability of glass to withstand these stresses. Non-strengthened glass materials have relatively low tensile strength yet high compressive strength. Therefore, most glass breakage is due to tensile stress failure. Mechanical properties are measured in a variety of ways: Modulus of Rupture (MOR) test measures the bending or flexural strength; shear modulus measures the amount of shearing or twisting forces a glass can withstand; Knoop Hardness Number (KHN) measures the hardness of glass; density is the mass value per unit of volume specific gravity is the ratio of the density of the glass to the density of water. (Carty & Senapati, 1998)

2.5.3. PHYSICAL PROPERTIESP

Regarding its physical properties, some glasses need a temperature of 650°C (1202°F) to melt and others need up to 1650°C (3002°F). Its average density is 2500kg/m3 but varies depending on the type of materials that make up the different types of glass. Its relative density ranges from 2 to 8 times the density of water, which makes it sometimes lighter than aluminum (relative density 2.7) but sometimes heavier than steel (relative density 7.85). Glass is malleable material through different methods that allow a different finish to be achieved, such as tempered, annealed, thermo-acoustic, armored, laminated glass, among others. The texture it obtains varies depending on the melting process and the materials with which it is made (Brigatti & Galán, 2013).

CHAPTER THREE

3. Conclusion

At the end of this research, we investigate that recycling and reprocess of such wastes is essential for environmental protection and maximizing economic benefits. And reemployment of waste as a raw material to produce the same, or very similar product, The transformation of glass to form glass-ceramic is called crystallization, also known as devitrification.Glass-Ceramic Production Routes is done in such methods :Conventional Method (two-stage), Modified Conventional Method (single-stage), PETRURGIC METHOD, Powder Methods So far only glasses produced from the molten state have been considered but in the last decades there has been considerable interest in using sol-gel and colloidal techniques to obtain the precursor glass in either powder or bulk form .Glass-ceramics produced by all the previous routes could be produced by sol-gel technique except that; they can't be manufactured from waste materials.

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