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MOULDS RECIPE SECRETS

Searching for transparency

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MOULDS RECIPE SECRETS. Searching for transparency

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Abstract

One of the main challenges on the creation of glass pieces in casting, is to obtain a surface of the glass as clean as possible, without adhering residues, or devitrification. In glass Sculptures where the surface is not flat, the polishing process becomes very complicated, and without having easy access to other polishing methods such as fire polish or acid polishing, it is difficult achieving pieces with a good level of transparency, making the process more complex and long.

Several variables determine the final appearance of the piece. In this work, one of the many variables is explored, which is the mold that will contain the molten glass. Normally the pieces made with this technique require subsequent treatments as they present a matte finish as a result of contact with the mold. The objective of this work is to investigate how to obtain greater transparency in the pieces by modifying the basic Investment, reducing time, effort, and costs in the realization of the glass pieces. Different additives in different proportions were added to the base mixture of silica and plaster, additives such as kaolin, graphite, ludo (recycled mold material), alumina, and talc. Three different glass compositions were used at a temperature of 850 ºC, for the casting. After the samples been cast the transparency were analyzed by visual comparison, and by measuring the transmittance of light, using as a comparison the base formula of 50% plaster and 50% silica. Three mold formulations resulted with the greatest effect on the improvement of the transparency of glass, using kaolin, graphite and ludo. The thermal properties of the mold using these 3 additives were analyzed using dilatometry and differential scanning calorimetry, showing that the 3 in different proportions may improve the thermal resistance of the moulds. Next, the effect on the transparency of the casting pieces by pre-drying the mold as well as the use of Shellac was analyzed, As a result, it could be determined that the presence of kaolin or graphite considerably improves the transparency in the pieces, as well as the previous drying of the mold at 94 ºC. The Ludo improves the appearance of the pieces in a lower proportion than graphite and kaolin but reduces the costs in the moulds materials, and the increasing of the strength and thermal resistance of the moulds. The results obtained have allowed me to generate pieces with sufficient transparency without the need for additional polishing processes.

KEYWORDS: Glass casting, moulds, transparency, sculpture, Thermal properties

Resumo

Um dos principais desafios na criação de peças de vidro em fundição, é obter uma superfície do vidro o mais limpa possível, sem resíduos aderentes, ou desvitrificação. Nas esculturas de vidro onde a superfície não é plana, o processo de polimento torna-se mais complicado, e sem ter fácil acesso a outros métodos como polimento a fogo ou polimento ácido, é difícil conseguir peças com um bom nível de transparência, tornando o processo mais complexo e longo.

São várias variáveis que determinam o aspecto final da peça. Neste trabalho, uma das variáveis foi tratada, que é o molde que vai conter o vidro fundido. Normalmente as peças confeccionadas com esta técnica requerem tratamentos posteriores, pois apresentam acabamento fosco em contato com o molde. O objetivo deste trabalho é investigar como obter maior transparência nas peças alterando o Investimento básico, reduzindo tempo, esforço e custos na realização das peças de vidro. Após a moldagem das amostras, a transparência foi analisada por comparação visual e pela medição dos espectros de transmitância, utilizando como comparação a fórmula base de 50% de gesso e 50% de sílica, selecionando como resultado três dos aditivos, caulino, grafite e ludo, que na parte experimental apresentaram melhores resultados relativamente à transparência do vidro,. As propriedades térmicas dos moldes com estes três aditivos foi analisada por dilatometria e calorimetria diferencial de varrimento, constatando-se que todos em diferentes proporções melhoram a resistência térmica dos moldes. Em seguida, foi analisado o efeito na transparência das peças fundidas pela pré-secagem do molde bem como pelo uso de goma-laca, como resultado, pôde-se constatar que a presença de caulino ou grafite melhora consideravelmente a transparência nas peças, bem como a secagem prévia do molde a 94 ºC. O Ludo melhora o aspecto das peças em menor proporção que o grafite e o caulino mas reduz os custos nos materiais, e no aumento da resistência e resistência térmica dos moldes. Os resultados obtidos me permitiram gerar peças com transparência suficiente sem a necessidade de processos de polimento adicionais.

PALAVRAS-CHAVE: Fundição de vidro, moldes, transparência, escultura, propriedades térmicas

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1. Introduction

Since I left Venezuela, my native country, my life trajectory has taken me along so many different paths, each decision has represented a change, new experiences and challenges, which, when viewed in retrospect, has allowed me to grow as a person in a way that I could never have foreseen. Fear, Uncertainty, Enthusiasm, Passion, Hope, are the keywords that have marked my path since then. Within all these changes (lifestyle, country, relationships, friendships, livelihoods), a few things have remained stable over the years, my family, my desire for spiritual growth and glass.

I discovered glass in Venezuela, more than 10 years ago and the fascination for the material has accompanied me ever since. All the changes and metamorphoses that I have gone through in my life are also reflected in my artistic work. My interest then turned to experimenting with new techniques, discovering the secrets of the material, attempting to understand it, and thus being able to reflect my internal world with it. This path has not been easy, coming from a world of numbers and finances, finding my own voice has been a titanic task and to be honest, I believe that this process is not over and that it will not end in the short or medium term, if at all ever ends.

Different voices told me that my work has to have a common thread, an expression that can be identified, and it is then when meditating I realized that my expression through art is similar to my lifestyle, I like to discover, experiment, and change. The engineer in me is still present, and research is part of my expression, and this is how there is a mixture of styles in me, tending sometimes to be figurative, sometimes tending to be geometric, sometimes organic, and who knows if in the whole process that I still have to go through, my form of expression will undergo any other change, but being honest, I like change, I like challenges, so it´s completely welcome.

My world is a world of search, of exploration, a journey to find myself, to find what moves me, what motivates me, it is the search for acceptance, mainly my own, and this leads me to investigate trying to find a balance between my analytical mind and my emotional mind. Debating between how I like to live and how I would like to live, trying to break my fears, my chains. In my work you can see that internal struggle, bodies that mix with each other imbued in a mixture of thoughts and emotions showing chaos but within the chaos an order.

Despite the different expressions in my work, there are elements in common, I like the Repetition. I find it very interesting to compose a piece made up of smaller parts, where the whole is configured by the sum of the parts. Various artists from different areas have been sources of inspiration in my current work, in glass artists such as Shayna Leib which created art pieces using glass cane (figure 1), Colin Reed which uses optical glass where transparency and opacity are combined in a single piece giving rise to incredible pieces (figure 2), and in other areas artists such as Matthieu Bourdel (figure 3), with his collages made mainly with photos of faces, in ceramics one of my favorite ceramists Zemer Peled with his intricate sculptures made up of small pieces of ceramics (figure 4), and in modern painting the beautiful work of Gustav Klimt (figure 5).

Figure 1 Glass, Shay naleib2011. https://shaynaleib.com/

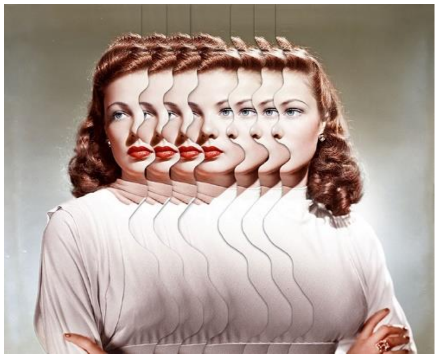

Figure 2 Duplicity', Matthieu Bourdel ,2014http://thereart.ro/matthieu-bourel-works/

Figure 3 Casa Forestal, Colin Reed Cast optical Glass, 2019 https://www.colinreidglass.co.uk/

Figure 4 White Dreams 1, Zemer Peled *,porcelain shards, fired clay, 2017,https://www.zemerpeled.com/*

Figure 5. The Kiss, Gustav Klimt 1908-1909, The Kiss Gustav Klimt 1908-1909

I tested and experimented with many thoughts through making art; recycling glass that I found on the street, which in turn came to represent those streets or places that I visited during my stay in the countries in which I have lived.One example can be seen in Figure 6, where I used the fusing technique combined with enamel painting, to recreate using small pieces of glass an aerial view of streets and buildings in cities.

Figure 6. Calles de Caracas, Gregorik Bermudez 2018. Glass Fusing. 50 x 50 x 2.5 cm

In several works I also used reverse painting with oil on pieces inspired by stones that I photographed in different places that I have visited, using a style inspired by the Byzantine mosaics that I got to know during my stay in Turkey, where I was also influenced by one of my favorite painters, Gustav Klimt (Fig. 7 and 8).

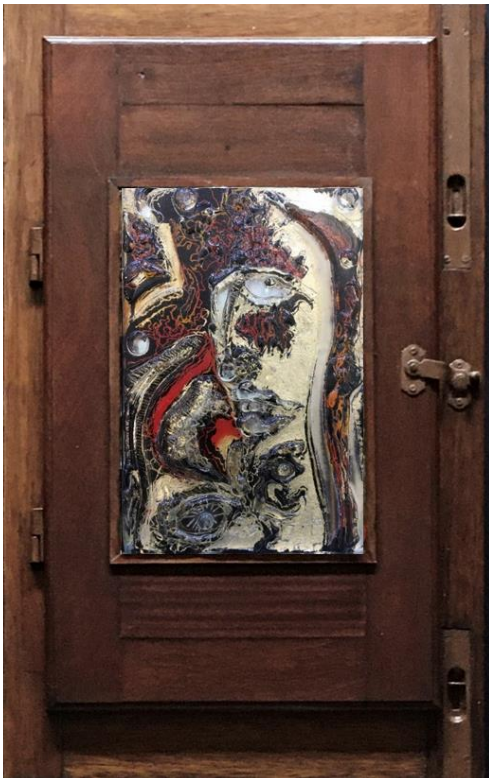

Figure 7. Mirada. Gregorik Bermudez 2018 Slumping, and gold reverse painting

Figure 8. *MyEve. Gregorik Bermudez Gilted Fused float glass, with reverse oild painted, on a restaured Wood window. 2020. 29 x 46 x 3.5 cm*

In sculpture my work mainly represents the human body where the parts are mixed in a kind of mosaic or collage, sometimes faces, sometimes dismembered bodies, sometimes elongated figures, representing interrelationships between people, the conversations between the various personalities within the being. In sculpture, I work principally with glass casting in which I like the combination of different textures. That is why I have a strong interest in achieving pieces with enough transparency to allow later play between transparencies and opacities. My testing and experimenting resulted in objects that represent my inner concerns (figure 9).

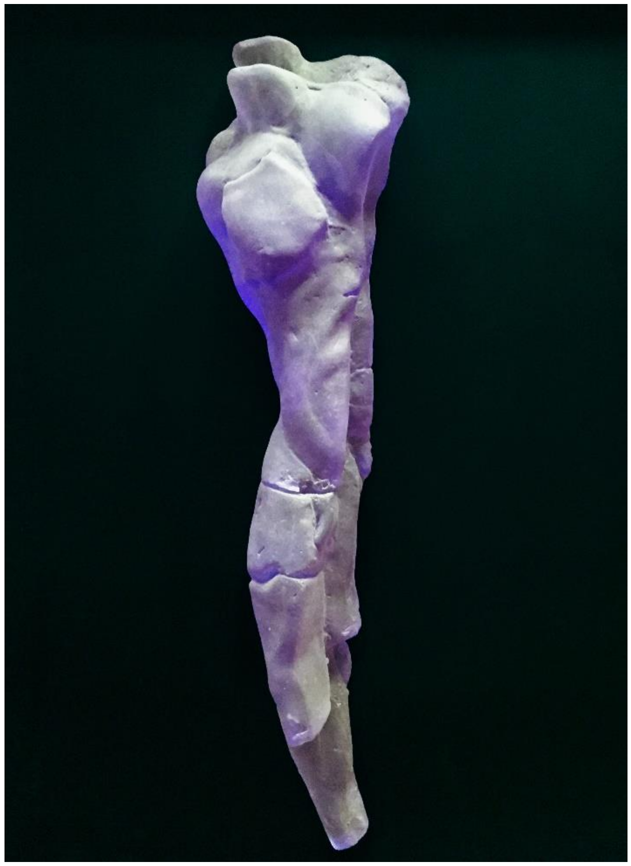

Figure 9. Encounters 1. Gregorik Bermudez. Kiln Casting 2020. 58 x 20 x 4 cm

It is interesting from my point of view that I like repetition in an environment where change is common. Working with glass I discovered that I like many different techniques, but one of my favorites is casting. I find fascinating how glass can transmit emotions and sensations through its surface, through brightness, transparency, textures, which generate different dimensions in the sculpture, which cannot be obtained with other materials. Glass casting offers numerous paths for exploration, some more challenging than others, but all with so much to investigate, so much to know, understand and learn. In the literature there is many information about Glass casting. Several authors like Angela Thwaites, Boyce Lundstrom and Lucartha Kohler, among many others, have written about it, and it is my objective when conducting this research, to understand one of the most important parts in the manufacture of this type of sculptures, which is the mold that will contain the molten glass during the cast, to achieve pieces with a Polished finish without requiring subsequent grinding or polishing processes, using materials that are easy to access and at the best possible cost. It is with this objective in mind that I begin this investigation, deepening the technique, in order to define the specific objectives to be investigated.

2

Chapter 2: Knowing About Glass Casting.

2.1. Kiln Casting. General Aspects

Glass casting is an ancient technique that dates to the times of the Assyrians and Egyptians[1]and has been passed down to the present day with relatively little change in its base concept, where moulds are used to melt and shape the glass. It is a technique where the glass is molded by using a refractory mold where the glass will melt, taking on the shape of the mold, where it will ultimately solidify, after a controlled cooling process[2]. The final result will be a piece of glass with a relatively matte appearance on its surface when compared to more familiar glass such as that made through blowing techniques like tableware or container glass. Making a casting is a very long process, and it requires that the artist undertake many aspects into consideration to achieve the desired effect on the surface. The appearance of glass casting pieces can vary from a subtle satin finish to a stone-like appearance. The appearance of the piece varies depending on different factors, and apart from the effects that can be achieved by cold working it; its appearance depends on factors such as the type of glass, the size of the grain used, the technique used, the fusing cycle, and finally the type of mold used[3]. The mate appearance is a product of the contact of the materials of the mold with the glass and in which, depending on the final finish that is desired, it will be necessary to carry out cold processes to polish or correct surface faults. In figure 10 it can be seen a piece made by the artist Colin Reed which was polished to achieve the presented effect.

The casting process can be done in hours or months, depending on the size of the piece, an example is in the manufacture of the Hale telescope mirror that lasted several months of cooling[4]. The working temperature commonly varies between 787 ˚C - 850 ˚C[5], depending on the type of glass used . It is a technique that requires a complex procedure, extensive knowledge of glass and that many times fails however besides the difficulties, it can also generate pieces with a high degree of beauty.

[1] El vidrio en el mundo antiguo. Marcelo Vigil Pascual. P 19
[2] O vidro como material plástico: transparência, luz, cor e expressão. Teresa Maria Castro de Almeida p191
[3] Lucartha Kohler. (1998). Glass An Artist´s Medium P 55, 142
[4] https://www.ecured.cu/Telescopio_Hale
[5] Henry Halem. (2006). Glass Notes. A reference for the glass Artist p121

Figure 10: Casa Forestal, Colin reed 2019. Kiln-cast and polished optical glass on a slate base. (https://www.adriansassoon.com/contemporary/casa-forestal-2019-2/)

Many factors can determine the success or failure in the making of cast glass; the type of glass used, the correct programming of the temperature curves for the melting and cooling of the piece, and the correct use of the refractory moulds where the glass will melt[6]. It is precisely this last topic that will be the object of this work, in which we will study the components of the moulds mentioned in the literature or used by glass artists to analyze if there is any correlation between the components of the mixture and the final transparency of the piece. But before that, we are going to explore the origins of this technique and how it evolved to our days.

2.2. Glass Casting in history.

The technique of glass molding is believed to have been originated in the molding of metal and ceramics, using the same systems that were used to make molten metal or terracotta figures within a mold, and dates back at least to the mid of the third millennium before the Common Era[7]. The appearance of glass could

[6] Lucartha Kohler. (1998). Glass An Artist´s Medium p55
[7] El vidrio en el mundo antiguo. Marcelo Vigil Pascual. P 18

have had its origin then, as a consequence of the process of obtaining and processing metals, which in turn requires the participation of ceramic materials.[8]

The processes consisted of pressing the molten glass into a negative mold or placing a mold in the furnace containing crushed glass. This technique was thus used to generate ornaments for jewelry, clothing and furniture, finding this type of object in Egypt, Asia, and Mycenaean Greece.

In Egypt, in the middle of the second millennium, during the 18th dynasty, this technique was used to make bowls, inlays and amulets, melting the glass inside a mold, the sand core technique was also used to make containers for unguents or perfumes[9] (See figures 11 -13).

Figure 11. Egyptian glass bowl, 18th Dynasty, reign of Amenthotep II (c. 1427-1400 BC). EA 36342 (wikimedia, 2018).

Figure 12. Tutankhamun's-Blue-Glass-&-Gold-Headrest Cairo Museum (Carolyn McDowall, 2011-2013)

Figure 13 Portrait Inlay of Pharaoh Akhenaten https://www.cmog.org/artwork/portrait-inlay-pharaoh-akhenaten

[8] El vidrio en la civilización egipcia. Descripción de piezas de vidrio del tipo incrustaciones de una colección de la Universidad de Sevilla. Boletín de la sociedad española de Cerámica y Vidrio. P 224

[9] El vidrio en el mundo antiguo. Marcelo Vigil Pascual. P 18

The use of lost wax appears in the Hellenistic age, used to make vessels of more complex shapes, normally colorless pieces[10]. Once molded, the glass was cold polished, or reheated to achieve a shiny surface. The technique continues in Alexandria manufacturing mosaic glass vases and passes to the West in the first half of the 1st century AD[11]., but with the appearance of blown glass, and blown in moulds it decreases its use for the creation of glasses, continuing for the creation of Diatretas, which production process in still under study, however, it seems likely that for some part of the production phase, moulds were used. (figure 13), and rib bowls (Figure 14).

Figure 14. Exquisite glass cage cup from Cologne, dated to the mid-4th century AD Collection Staatliche Antikensammlung. (Carolyn McDowall, 2011-2013)

During the Roman period, the technique is still used to create colorless glass that would later be finished with other techniques, for the creation of Diatretas[11]. The mold consisted of two or more interconnected parts where the glass was added in the form of frits or incorporating the molten glass directly to the mold. Italy is believed to be the source of the majority of the early polychrome imperial glass cast in casting, while monochrome casting was more prevalent elsewhere in the Mediterranean[12.]

[10] El vidrio en el mundo antiguo. Marcelo Vigil Pascual. P 20
[11] El vidrio en el mundo antiguo. Marcelo Vigil Pascual. P 81-82
[12] Price, J., A survey of the Hellenistic and early Roman vessel glass found on the Unexplored Mansion Site at Knossos in Crete, in Annales du 11e Congres. 1990: Amsterdam.

The technique was also used during this time to create mosaic or Mirafiori glass vessels. During the Christian era, they continued to use moulds for blowing, and pieces made in moulds directly, an example of this are the called rib bowls.

Figure 15 Roman glass rib bowl[13].

The forms became more limited in the late first century but continued in production through the second or third decade of the second century. Faded vessels and bowls spread throughout the Roman world at the end of the 1st century and the beginning of the 2nd BC[14]. It is in the 19th century, in the framework of the industrial revolution, that a revitalization of the technique is observed, known generically as kiln-forming. "The modern studio glass movement" is developed by the French Pate-de-Verre artists, artists such as Decorchement, Argy-Rousseau and Amalric Walter, who developed an impressive skill in creating cast pieces with colored glass (Figure 15), a technique that was, for a long time, lost with the death of these artists[15].

Figure 16. Poisson art Deco, 1930 Almaric Walter (wikipedia, 2019)

[13] *https://www.arqueologiaclasica.com/obras-de-arte/cuenco-romano-en-vidrio-con-decoracion-gallonada*
[14] El vidrio en el mundo antiguo. Marcelo Vigil Pascual.
[15] Contemporary- Kiln Formed glass. Keith Cummings 2009. P27-33

This was the time of the Second World War so there are no written records of the technique. The color palette was created by the artists themselves, by obtaining fine grains of glass mixed with oxides, applying them directly to the mold or, as in the case of Walter, applying it with a brush as if it was a painting. [16]

Gradually the secrets have been discovered through the research of many contemporary artists. The main differences are the use of standard glasses, and the increasing use of casting specialists who perform the casting process on the artists' pieces, creating large-scale studies[17].

Since then, a wide range of processes, in use at the present time, has been developed, under the framework of the studio movement. The possibilities of the material have been extended thanks to technological advances, to a level such that there are no previous equivalences, with processes that have allowed a wide range of possibilities for the artist to express himself through his art[18].

2.3. Description of the technique

We can find many different technical approaches framed within the kiln casting umbrella. These vary in the final finish and shapes, but all have in common the use of refractory moulds for its realization and the use of a furnace as a heat source to melt the glass, which can be used in different forms as dust, frits, chunks, rod form and also can be pore as a molten glass (Figure17)[19].

[16] Contemporary- Kiln Formed glass. Keith Cummings 2009. P20
[17] Contemporary- Kiln Formed glass. Keith Cummings 2009. P21
[18] Contemporary- Kiln Formed glass. Keith Cummings 2009. P61
[19] Glass an Artist Medium. Lucartha Kohler. Krause publications (1998). p60

.

GLASS CHUNK

GLASS DUST

FRIT

GLASS CULLET

GLASS ROD

MOLTEN GLASS

Figure 17. Different forms of glass used for casting pieces.

Kiln Casting can be classified according to the type of glass, as well as the type of mold used[20]. Some of the techniques are shown below:

Pâte de Verre: This technique uses fine transparent or colored glass grains in a paste that is generated by mixing the glass with water and a binder, this paste is applied inside the mold, with a brush, trowel, or fingers, according to the desired design, and then fused in the kiln. The result is a matte piece, with an

[20] Mould Making for Glass. Angela Thwaites. A&C Black London (1988). P 15-19

internal shine, coming from the low temperature at which the process is carried out, between 704-800 ºC depending on the kind of glass and the surface looks chossed by the artist.

Frit Casting: method similar to *Pâte-de-Verre* where it differs mainly is in the size of the glass used, the larger the frit size, the clearer the glass obtained in the final piece.

Fuse casting: where large pieces of glass are used, and it is the technique that generates greater transparency. Large pieces begin to flow at a lower temperature. "It seems that when glass is moving or flowing it doesn't devitrify as readily".[21]

Open moulds: It is one of the easiest kiln casting technique. As its name suggested, the mold is open in the upper part, havind the figure, thatit is wanted to be cast, in the botton, Generating a glass piece with a flat base, the piece is easy to remove from the mold, allowing the creation of large pieces.

Lost wax (cire perdue): characterized by the creation of closed moulds of pieces that can have undercuts or intricate shapes, a process that allows multiple copies to be made by using a previous mold where the wax will be introduced generating the model from where a final mold is created. Once the mold is made, the wax is removed by using steam, and the mold is ready to place the glass and be putted in the oven.

Core casting: method used to make hollow objects, with an internal space where one shape appears to be floating or suspended within another. It is possibly the most complex process within the kiln -forming methods[22], The mold has, in addition to the walls, a three-dimensional figure inside, which is surrounded by the molten glass, obtaining a piece with a hole in the shape of the inner piece.**Drip Casting**: process in which a ceramic pot is placed on the top of our mold and the glass flows from there when the temperature increases, produces high transparency.

2.4. Aspects to consider when making the mold

When we are creating glass pieces using moulds, there are many important aspects to take care of. One of the most important aspects is in addition to the knowledge of how to make the mold itself, to understand how the different components that make up a mold will affect the final result of the glass pieces. Casting is a glass sculptural technique where a mold is generated in most cases with Plaster as the main component, and where the glass is melted to be adapted to the shape and then generate the final piece.

Several books and publications have been written about how to build a mold and the different recipes to make it. Some of them have been used as a reference for this thesis and are listed in the bibliography. For the construction of an effective cast for glass casting, we must consider different variables:

Variables that affect the mold[23]:

[21] Glass an Artist Medium. Lucartha Kohler. Krause publications (1998). P 61
[22] Mould Making for Glass. Angela Thwaites. A&C Black London (1988). P 15
[23] Boyce Lundstrom. (1989). Glass Casting and Mold making. Canada: Vitreous Publications

1) **Homogeneity of the Investment mixture**: The ideal Plaster mixture is one in which the particles are homogeneously dispersed in the water in such a way that they produce an equally homogeneous mixture.
2) **Purity and the temperature of the water used:** Both variables affect the setting time of the mold as well as the final surface, according with Lundstrom[24] it is recommended to use potable water with no impurities, that prevent havig particles that can affect the setting time, and at a preparation temperature between 21 and 23 degrees Celsius, higher the temperature, faster the setting time.
3) **Proportion of water versus Plaster**. This proportion affects the strength of the mold and therefore its behavior. The addition of additives in the mixture should be considered when modifying the amount of water. In the case of materials that absorb it, water should be added in more quantity than it is used for plaster and silica investment
4) **Soaking the mixture**. The mixture must be completely saturated with water before starting mixing so it is recommended to wait between 2 and 4 minutes. Less time will affect the quality of the final mold.
5) **The Mixed**. This is the most important process for the production of a quality mold, the longer the mixing time, the greater the strength of the mold, but it will affect the porosity of the mold which is also a very important factor, it is necessary for glass moulds that exist a certain degree of porosity to facilitate the release of water without generating cracks, for It is recommended to soak the Plaster, then mix it for 2 or 3 minutes and finally using an electric mixer for 2 or 5 minutes.
6) **Cleaning of the mixing container**. The existence of Plaster remains in the containers or removers will affect the setting speed, accelerating the process.
7) **Materials used to create the mold**. There are many formulas in the literature, so in order to obtain the best effect, it is necessary to understand how to use them. In that sense, it is important to understand the purpose of each raw material used, as well as to understand its effect on the final piece of glass.

Depending on the materials used as investment, the moulds will exhibit different behaviors[25] (figure 18), such as:

a) Be able to take and retain the base form, and allows digging or sculpting details, if it’s necessary.
b) Be able to maintain strength and integrity during the firing cycle c) It should be easily removable from the glass without damaging it, and give a good surface appearance in the final glass piece[26].
d) More eco-friendly as possible
e) Easy to make and use
f) Economically effective

[24] Glass Casting and Moldmaking, Boyce Lundstrom, P 57
[25] Henry Halem. (2006). Glass Notes. A reference for the glass Artist. USA. P62
[26] Mould making for glass, Angela Thwaites. A&C Blaxck, London. P21

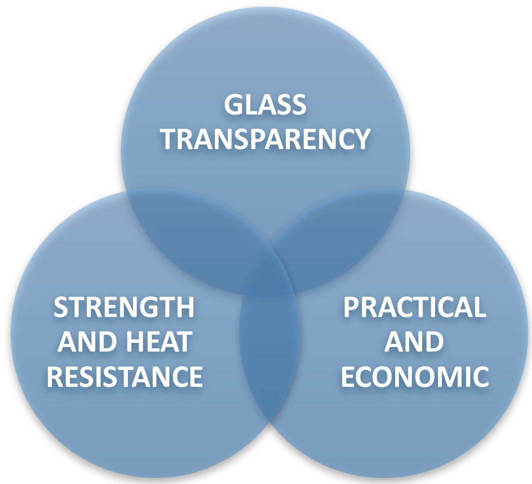

Figure 18. Important Characteristics to be obtained from a mold investment

2.5. Casting Problems.

Several complications can be generated when a piece is made with the casting technique, as spikes on edge because the glass is trapped on the mold walls, not wanted air bubbles, opacification of the casting piece[27], mold material failure generating cracks, fracture of the piece due to incorrect firing cycle [28], unwanted inclusions, excess of glass that generates overflow, insufficient glass which generates failures in achieving the desired shape, loss of details in the figure due to trapped air due to the incorrect use of vents, stress due to incompatibility in the glass, etc. Among all of them described problems, we are going to focus on the emerging of glass opacity due to the mold.

When creating pieces with Glass Casting, the surface in contact with the mold material, generally has an appearance that ranges from a subtle satin finish to a sugary appearance, product of the formation of crystals on its surface because of devitrification[29], and depending on the materials of the mold, residues of the mold may also stick to the glass [30] . In figure 18, both issues can be observed, the devitrification in the edges of the glass, and fractures originated in the mold that is reflected in the final piece.

If we aim to obtain a transparent surface on the glass piece, it is necessary to make an extra effort by doing a cold working, to give it the desired finish looks. This generates a loss of time, additional costs, and greater effort, and additionally the risk of damaging the piece. This is why the concern arises to find ways to avoid cold working and obtain a transparent appearance in the pieces, by changing the moulds properties of the mold.

[27] Troubleshooting For Glass Art PROBLEM - CAUSE – CORRECTION. Dennis Brady Glass Campus Publishing
[28] Kiln casting: What can go wrong and how to fix it? Heike Brachlow. 2019
[29] El vidrio. Jose Maria Fernandez Navarro. Third edition. Artegraf,S.A. P 264
[30] Angela Thwaites. (2011). Mould Making for glass. P26

Figure 19. Defects obtained from an incorrect glass mold. Pictures from glass manise.com

As devitrification is the main cause of the opaque appearance on the glass surface, let's understand more about it.

2.5.1. Devitrification.

The general term "devitrification" is intended to indicate the natural tendency of a vitreous system, to evolve towards a state of higher structural order such as crystal[31]. In the case of glass, it is called the solid inclusions born in the glassy mass by crystallization of one or more of its components. It is governed by two processes that run in parallel with a lag in its variation with temperature, the process of formation of crystalline nuclei and the process of crystal growth.[31]

The variables that determine the appearance of crystals are the speed of both processes, which depends on the temperature of the glass, the composition, the atmosphere in contact with the glass, the presence of solid particles that serve as nucleation seeds, and the interfaces, where devitrifications may occur between the glass and the walls of the refractory material, the heterogeneity of the surface reduces the energy needed to start the nucleation process, in addition to a chemical aspect where the components of the refractory dissolve in the melt generating nucleation seeds[30].

According to the external and internal factors involved in the process, this can be defined as homogeneous or heterogeneous.In the homogeneous case, the crystal that is formed has a composition completely similar to that of the vitreous liquid; while heterogeneous nucleation is rather deliberately caused by the addition of nucleating agents (particular oxides, impurities, etc.).

It is important to understand, how the mold reacts with the molten glass to minimize the defects that can be obtained in the final piece. For that it is necessary to understand the mold composition which is what it will

[31] El vidrio. Jose Maria Fernandez Navarro. Third edition. Artegraf,S.A. P 264

be studied next, focusing on investment recipes found in the literature, and on those components that allow the generation of greater transparency in the glass.

2.6. Materials Used In the Construction of Casting Moulds

The materials that are used for the formation of refractory moulds for glass can be grouped into 3 groups, Binders, Refractories and Modifiers depending on the role assumed by each of them. A summary can be observed in table 1.

TABLE 1: COMMON MATERIALS USED FOR CASTING MOULDS. [32]

BINDERS / CEMENTS	REFRACTORIES	MODIFIERS
Gypsum Plaster	Silica	Vermiculite
Hydroperm	Diatomite	Pearlite
Hydrocal cement	Hydrated Alumina	Grog
Portland cement	Zirconia	Sawdust
Calcium Alumina cement (fondu)	Olivine sand	Air (soap)
	Silica sand	Alumina fiber
Colloidal Alumina	Grog	Ludo
Colloidal Silica	Ludo	Kaolin clay
Clay (fire clays)	Molochite	Shell Prymer
	Siicon carbite	Talc
	Diatomatious Earth	

2.6.1. Binders:

Also called cements are those that maintain the structure of the mold, which keep the refractory particles in place. Different binders have different working temperatures, so depending on the use given to the mold we must choose the most suitable. Binders maintain their bond strength in 3 different ways[33]: Hydraulic Bond (creation of crystals by hydration); Molecular Attraction (van der Wall), and Ceramic Bond or glassification (the mechanical strength is developed by heating earthy materials and thus producing glass or effective crystallization)[34].

For this study we are going to use the gypsum Plaster as a Binder, for being the most common among casting artists, it is easy to acquire, and economic[35].Plaster, or Plaster of Paris or calcium sulfate ($CaSO_4.1/2$ H_2O), it is the most used binder for moulds in casting of glass, which is the result of grinding mineral gypsum, after it has been calcined by heating it to about 150 °C. It is a Hydraulic Bond Binder so by adding water the

[32] Glass Casting and Moldmaking, Boyce Lundstrom, P 67
[33] Glass Casting and Moldmaking, Boyce Lundstrom, P 45
[34] https://www.merriam-webster.com/dictionary/ceramic%20bond
[35] Mould making for glass, Angela Thwaites. A&C Blaxck, London. P25

Plaster rehydrates until it develops its full strength[36]. The Plaster begins to lose its strength in the kiln at 50 ºC, losing its structural stability between 700-840 ºC[37]. At the temperature of 649 ºC pure Plaster start to shrink radically causing cracks in the mold that is the reason it is necessary to use, materials that are inert to the temperature.

The minimum quantity necessary to keep the integrity of the mold is 50% in weight, also it is recommended to use different grain size of the material in order to obtain more consistency and therefore physical integrity. In table 2 we can see some of the relevant chemical characteristics.

2.6.2. Refractories

Refractories are materials that can withstand thermal change without deforming or changing chemically in an certain heat interval [39] (See table 2). Refractories have different properties to resist the corrosive action of hot glass, where some materials adhere more to glass than others.

TABLE 2 REFRACTORIES AND THEIR REACTIVITY WITH GLASS[33]

	Alumina	Silica	Grog	Ludo	Insulation brick	Molochite	Olivine sand	Zirconia
Glass Reactive	NO	YES	YES	+/-	No information	LOW	LOW	NO

For this work, Silica will be used as the main refractory, as it is the most commonly used refractory, and the effect of Alumina and Ludo using them in conjunction with Silica will be analyzed. These three refractories are chosen for their easy availability, leaving the rest for further investigation.

Silica (SiO_2 melting point of 1726 ° C**)**[38] is the most used refractory in the creation of moulds, although it is more reactive than Alumina and Ludo at high temperatures, which causes it to adhere to the glass surface[39]. It must be incorporated as flour (200 mesh and finer). In its natural state, it can be found in different crystalline forms, between then quartz, topaz and amethyst. Silica also goes through quartz inversion at approximately 593 ºC so it must be heated or cooled carefully at that temperature to avoid the generation of cracks. Silica, it is reactive with glass at casting temperatures, exchanging iones, thus it adheres to glass.

Alumina (Hydrated):Alumina (Al_2O_3 Melting point: 2702 ° C)[40] comes in different forms of crystals that despite having the same chemical formula react differently to heat. Its resistance to thermal shock makes it an excellent choice for open faced moulds that are intended to be used for many reproductions, linked with

[36] Mould making for glass, Angela Thwaites. A&C Blaxck, London. P26
[37] Mould making for glass, Angela Thwaites. A&C Blaxck, London. P27
[38] Softening And Melting Of Sio2, An Important Parameter For Reactions With Quartz In Si Production, Eli Ringdalen, , Merete Tangstad2, SINTEF Materials and Chemistry University of Science and Technology, Trondheim, Norway
[39] Glass Casting and Moldmaking, Boyce Lundstrom, P 49
[40] Pradyot Patnaik. *Handbook of Inorganic Chemicals*. McGraw-Hill, 2002, ISBN 0070494398

Silica colloidal allows to generate very strong moulds with low thermal expansion. Alumina is not reactive with glass, and in theory it is good for glass moulds, it increases the strength and density of the glass[41]

Luto or Ludo: crushed previously fired mold material, which has lost both physical and chemical water during burning. It can act both as a refractory material and as a modifier. Using Ludo is a good practice to cut costs, being able to reach up to 50% of the total dry material of the mold. Its use increases the size of the particles of the mixture, increasing strength and reducing cracking. It is easier to remove from the mix as it sticks less to the glass. Ludo makes the mold less likely to crack due to fast kiln drying.[42]

2.6.3. Modifiers

Some materials are added to the mold mixture to improve drying time, increase porosity, or counteract the expansion or contraction of other materials, and to facilitate the separation of the glass from the mold. They usually weaken the structure but adding in small quantities avoids weakening the mold. In table 3 it can be seen some of them which are indicated in the literature as elements that influence the finish of the glass, and in table 6 it can be seen some of their properties. In this study, we are going to use Kaolin, Graphite and Talc as modifiers.

TABLE 3 MODIFIERS AND THEIR REACTIVITY WITH GLASS[43]

	Kaolin	Talc	Vermiculite	Carbon or Graphite
Glass Reactive	NO	NO	Yes	No information

Kaolin or china clay is a white-firing clay. It has low plasticity and high refractory qualities and contains 38% -39% of Alumina[42]. It prevents mold mix from sticking to the glass, as an addition of 3 – 5% helps to separate the glass from the Plaster, however, more than 10% can promote devitrification.

Talc: also known as soapstone or steatite, is a phyllosilicate mineral with chemical composition $Mg_3Si_4O_{10}(OH)_2$. Talc has refractory properties, being inert up to 850 ºC[44]The addition of talc makes the mold a bit softer with the idea of having less potential for glass cracking as it *contracts*. Talc is documented in some French recipes from the 19th century, and is still used by some practitioners with a use similar to Kaolin.[44]

Graphite: Graphite is a mineral, one of the two more common allotropes of carbon. Due to its high point of fusion, it also has applications as a refractive material, and in glass it's used as separator between the mold and the glass. Graphite is an element used as a release agent in sand casting, and is also used in the form of Graphite moulds in the direct casting of the glass (poring molten glass on the mold)[45]. Besides in the literature, I was not able to find, the use of Graphite as an investment material for casting, their applications

[41] Pradyot Patnaik. *Handbook of Inorganic Chemicals*. McGraw-Hill, 2002, ISBN 0070494398
[42] Angela Thwaites. (2011). Mould Making for glass. P 27- 29
[43] Mould making for glass, Angela Thwaites. A&C Blaxck, London. P 30-33
[44] Angela Thwaites. (2011). Mould Making for glass P 31
[45] Lucartha Kohler. (1998). Glass An Artist´s Medium P 35

suggest that it can be a useful raw material in the mold production. Even Keith Cummings in his book Contemporary Kiln-Formed Glass, page 195, mentions "*An area for experimenting is the addition of a small amount of Graphite to the Surface mold mix*"[46].

2.7. Other Considerations to Achieve Transparency

In the literature, shellac it is used for artists, including Almaric Walter, on the surface of the mold to reduce the amount of material that can adhere to the glass, obtaining a smoother surface, as well as to stabilize the surface of the mold avoiding that dust contaminations in the glass.[47] nother aspect mentioned in the literature of great importance is the pre-drying of the mold, a process that reduces the adherence of material from the mold to the glass[47].

Shellac: Shellac is a resin secreted by the female lac bug, on trees in the forests of India and Thailand. It is processed and sold as dry flakes (pictured) and dissolved in alcohol to make liquid shellac, which is used as a brush-on colorant, food glaze and wood finish. Shellac functions as a tough natural primer, sanding sealant, tannin-blocker, odour-blocker, stain, and high-gloss varnish..[48] Some artist use this material in order to dismiss the quantity of material attached to the glass.

2.8. Formulation for Moulds Investment in the Literature

In the literature, it can be found various formulations, suggested by different artists, product of their personal research. Artists as Ann Robinson, Angela Thwaites, Lucartha Kohler, Boyce Lundsttrom, Ekrem Kula, among others who have shared their investment formulations in books or with their students. Their recipes are adapted to different needs, whether greater resistance at higher temperatures, higher resistance to the pressure of the molten glass, or to obtain a better and cleaner surface. On the other hand, in the specialized glass market, there are already manufactured raw materials that offer different characteristics to the mold. Some of them can be seen in table 32, Annex 5. Those premixed materials usually have a higher cost than prepared the mix by yourself (see chapter 6.1, P 53), and sometimes can be difficult to obtain in your living area, so implies generating additional costs for shipping.

The search for already known formulations is essential in this research as a starting point . Table 4 shows a compendium of formulations obtained both in literature and in workshops previously done, and the corresponding references.

[46] Keith Cummings. (2009). Contemporary Kiln Lack of transparency on the glass surface -formed glass. London: &C Black Publishers
[47] Angela Thwaites. (2011). Mould Making for glass. P 27
[48] *Cambridge Dictionary*. Retrieved 19 October 2020.

TABLE 4 RECIPES FROM THE LITERATURE

INVESTMENT FORMULA	DESCRIPTION MAIN CHARACTERISTICS	AUTHOR
First layer 38,5% Plaster+38,5% Silica+19,2% fine Grog+3,8% shredded paper *Backup layer* 32,3% Plaster+32,3% Silica+10,8% fine Grog+21,5% course Grog+3,2% shredded paper	For large core vessels and large solid casting Strength	Ann Robinsons[49]
50% Plaster+50% Silica	Open faced, one-part molds	Boyce Lundstrom[50]
49,8% Plaster+49,8% Diatomatious earth+,5% shelf primer or EPK	Good release properties Temperature resistance 843 ºc	
79,8% hydroperm+19,8% Diatomatious earth+,5% EPK, or Kaolin clay	Surface quality Permeable Good details Temperature resistance 871 ºc	
55,6% hydroperm+22,2% hydrocal+22,2% Silica+,2 cups chopped fiber	All kiln-forming process Temperature resistance 885 ºc	
59.5% hydrocal+39.5% Olivine sand+1% EPK	Slump Re usable Temperature resistance 704 ºc	Boyce Lundstrom[51]
40% Plaster+40% Silica+20% Grog	Lost-vegetable casting and pate de Verre Strength	Jan Heins[26]
32% Plaster+32% Silica+32% Ludo+4% Kaolin	Simple shapes Lower shrinkage Temperature resistance Softer	Angela Thwaites[52]
33% Plaster+15% Silica+15% Olivine sand+33% Ludo+4% molochite	Strength	
33% Plaster+10% Silica+10% Olivine sand+33% Ludo+10% Grog+4% Kaolin	Strength Temperature resistance	

49 Henry Halem. (2006). Glass Notes. A reference for the glass Artist. P88
50 Boyce Lundstrom. (1989). Glass Casting and Moldmaking P 59-62
51 Boyce Lundstrom. (1989). Glass Casting and Moldmaking P 59-62
52 Angela Thwaites. (2011). Mould Making for glass P 99-101

INVESTMENT FORMULA	DESCRIPTION MAIN CHARACTERISTICS	AUTHOR
First layer 47,9% Plaster+46% Silica+6,1% Kaolin Second layer 48,1% Plaster+24% Silica+24% flint+3,8% Kaolin Third layer 50% Plaster+50% flint Fourth layer 33.3% Plaster+5.5% + Olivine sand+28% flint+33.3% Ludo	Fine surface	Angela Thwaites
28% Plaster+10% ground sand+18.5% Olivine sand+13% fiberglass +18.5% Silica+12% Kaolin	Fine surface	Argy-Rousseau
50 % Plaster - 35% Silica flour – 5% Kaolin– 1 % old boar – 9 % Ludo	This formula was given to me by the Turkish Professor and Artist, Ekrem Kula, during my studies at Anadolu University Fine surface Strength	Ekrem Kula

According to the information presented above, the plaster was selected as the main binder, as it is the most widespread binder used by artists, and is easy to obtain. Alumina is selected because it does not react with glass and Ludo because it is indicated as an element that reduces the adherence of the mold material to the glass, and reduces the molds costs.

Silica is selected as the refractory, as it is the most commonly used by artists, so it is interesting to know how to reduce its adherence to glass by using modifiers.Kaolin is selected as the modifier because it is the most widely used, as well as Graphite, whose effect is not documented as a modifier in the mold investment.

As part of the thesis experimentation, it is proposed to analyze the effect of each of the selected additives in the investment of the molds, to see the results on the glass surface. According to the information presented in table 7, the proportions of the selected additives will be used as a guide to define the proportions to be experimented on in the following stages of the thesis (see table 5).

TABLE 5 PROPORTIONS OF THE SELECTED COMPONENTS IN THE LITERATURE FORMULATIONS

MATERIAL	PROPORTIONS (%)
Plaster	32 - 50
Refractory	30 – 57.7
Kaolin or EPK	1 - 12
Ludo	9 - 33

2.9. Objectives

Framed in my main objective which is to understand one of the most important parts in the manufacture of the glass sculptures, which is the mold that will contain the molten glass during the cast, in order to achieve pieces with a more polished finish as possible without requiring subsequent grinding or polishing processes, using materials that are easy to access and at the best possible cost. After analyzing the information collected on the chapter, the specific objectives to investigate in this thesis are defined as

1. Analyze whether the combination of silica and Alumina as refractories brings greater transparency in the casting pieces.
2. To understand the effect of Kaolin, Talc, and Graphite as modifiers, in obtaining transparency and a clean surface.
3. To understand if the previous dry out of the mold, affect the transparency of the glass.
4. To understand if the use of Shellac, has an impact on the transparency of the pieces.
5. To analyze the impact of using Ludo (Reused Investment already fired) in mold mix as an element that will reduce the cost of the materials, and to understand if it has an impact on the glass transparency,

3

Chapter 3. Experimental part.

To meet the objectives set, it is necessary to obtain samples using the different elements previously selected. Molds will be created starting from the base formula of plaster + silica and analyzing the influence of each additive separately, varying the proportions of the mixture. Each formula will be tested with three different types of soda-lime silicate glasses, which I have been working during the master. Window glass (Float glass), Furnace glass, which is provided by Atlantis Company, and a glass I call Turkish glass provided by the Çaliskan Kristal Company. It is necessary to understand what material we are working with, so the first experiment to be carried out is the characterization of the glasses by calculating their composition, their expansion coefficient (COE), the glass transition temperature (tg) and softening temperature (ts).

3.1. Characterization of the glasses.

3.1.1. Composition

The composition of the three glasses will be obtained by X-ray fluorescence, using a portable μ-EDXRF spectrometer ArtTAX 800 of Intax GmbH. The spectrometer is equipped with a molybdenum target X-ray tube and an electrothermally cooled silicon drift detector. The system is coupled to a color CCD camera that provides a magnified digital image of the probed area. The primary X-ray beam diameter is approximately 70 nm and is focused by means of polycapillary X-ray optics. Spectra were acquired under the following conditions: voltage 40 kV, intensity 600 μA and 360 s of live time. For light element analysis, including aluminum, a helium purging system was used. Three measurements were taken in each sample and the average was calculated.

The elements of the composition will be compared with standard formulations of the 3 main types of glass to define in which group the glass used belongs, and the oxides presented in the glass will be compared according to the theoretical effect of each oxide, to understand the expected behavior respecting the fluidity and the tendency to devitrification.

3.1.2. Thermal Properties

The three types of glass are characterized by calculating the glass transition temperature (Tg), the softening point (ts) and its expansion coefficient (α), been determined by Dilatometry. The objective is to analyze if there is any correlation between these values and the transparency of the obtained glass samples. One sample from each glass composition, with a minimum length of 2.5 cm, and a diameter of approximately 0.5 cm, was analysed. A Netzsch Dil402PC dilatometer was used from 25 to 900 °C with a heating rate of 5

K·min^{-1}. The samples were placed in the dilatometer and the glass was heated until the 850ºC, calculating the previous mentioned parameters.

It would be expected that those glasses with a higher glass transition temperature would present a lower degree of devitrification, since it would be less reactive with the walls of the mold. Now when the float presents a tin layer on one of its surface this hypothesis can vary.

3.2. Glass Samples Preparation

3.2.1. Molds formulation

In this part of the investigation we are going to define the investment formulations for the molds where the glasses will be melt, and then be able to observe, after firing, their surface conditions. The molds will be done using a silica plus gypsum composition as the base element, adding one additive at a time in different proportions in weight. Once the molds have been made, they will be taken to the kiln at a maximum temperature of 850 Cº, and the visual results obtained will then be analyzed. The elements and proportions to be analyzed can be observed in the following tables.

TABLE 6 DEFINITION OF THE SAMPLES INDICATING ADDITIVE AND PROPORTION OF EACH COMPONENT

BASE FORMULA				It will be used as a comparative basis with respect to the other formulas
Proportion	Plaster %	Silica %	Additive %	
Sample 1	50	50	0	

LUDO				The objective of these experiments is to analyze the effect of the use of recycled material on the quality of the final glass. If the obtained glass does not vary, it would reduce costs in the manufacture of the molds.
Proportion	Plaster %	Silica %	Additive %	
Sample 2	45.0	45.0	10.0	
Sample 3	42.5	42.5	15.0	
Sample 4	40.0	40.0	20.0	

KAOLIN				Main component used on the formulations from literature for obtaining clean surfaces. (See point 2.6.3)
Proportion	Plaster %	Silica %	Additive %	
Sample 5	47.5	47.5	5.0	
Sample 6	45.0	45.0	10.0	
Sample 7	42.5	42.5	15.0	

TALCO				It is used as an Additive by some artists for obtaining clean surfaces. (See point 2.6.3)
Proportion	Plaster %	Silica %	Additive %	
Sample 8	47.5	47.5	5.0	
Sample 9	45.0	45.0	10.0	
Sample 10	42.5	42.5	15.0	

GRAPHITE				It is used as a separator in molds for sand casting, as well exist molds completely made of this material. suggested as a research area by *Keith Cummings* (See point 2.6.3)
Proportion	Plaster %	Silica %	Additive %	
Sample 11	47.5	47.5	5.0	
Sample 12	45.0	45.0	10.0	
Sample 13	42.5	42.5	15.0	

ALUMINA				It is used as an Additive and also as a former element. Linked with Silica colloidal allows generating very strong molds with low thermal expansion. Along with Kaolin it is used as separator for kiln shelf and molds[53].
Proportion	Plaster %	Silica %	Additive %	
Sample 14	47.5	47.5	5.0	
Sample 15	45.0	45.0	10.0	
Sample 16	42.5	42.5	15.0	

3.2.1.1. Firing Cycle

After making the molds, the casting will be carried out up to a temperature of 850ºC, The maximum temperature will remain constant in all samples. The Firing cycle can be seen in table 7, being calculated for a 1.5 cm thick sample. To reduce factors unrelated to the Firing process, the glasses were cleaned with alcohol to remove traces of grease, the same cycle and the same oven were used for the firing, in order to guarantee similar conditions in all samples and the mold investment were mixed with an electric stirrer to guarantee homogeneity. The ideal annealing cycle depends mainly on the thickness and the COE of the glass[54], being that the samples were made with three glasses of different composition, a fixed thickness was defined for the samples of approximately 1 cm, the cycle was calculated, for 1.5 cm samples thus lowering the cooling rate of the annealing and the cycle was adjusted to cover a wider annealig range between 550-443 ºC.

TABLE 7 FIRING CYCLE FOR 1.5 CM THICK GLASS

STEP	TIME (min)	TEMPERATURE (ºC)
0	**0**	**24**
1	**180**	**427**
2	**360**	**621**
3	**390**	**621**
4	**450**	**690**
5	**480**	**850**
6	**540**	**850**
7	**570**	**550**
8	**1170**	**443**
9	**1770**	**265**
10	**1950**	**24**

[53] http://glasstips.blogspot.com/2009/08/smooth-kiln-wash-on-shelves.html
[54] Lucartha Kohler. (1998). Glass An Artist´s Medium P 213

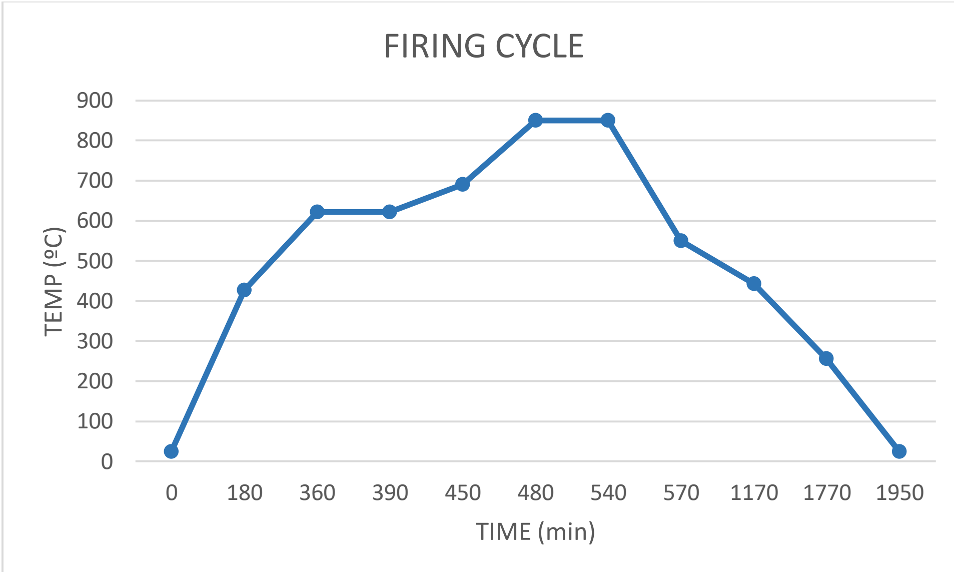

Figure 20: Graph with the firing cycle used

3.2.1.2. Selection of the Best Formulas

The procedure performed to order the samples, from the best results to to worst ones, is explained below. By having a different glass formulation, the formulas may react differently between one glass and others at the same temperature 850 ºC, but in this case, we are going to take an average between all the samples.

For each formula,two samples will be made per each type of glass, one with a textured surface, and the other smooth, giving a total of six samples per formula, giving a total of 36 samples. After obtaining the samples, a visual comparison is made between them, taking Sample 1 (50/50) as a basis for comparison. Sorting them from highest to lowest quality, considering quality as the absence of devitrification or residues adhered to the glass, and only using visual analysis, the best sample would be the one that has the least residue on the surface (white shading).

It will be calculated how many times the samples of each formula are located in the first, second, third or fourth position, scoring from 4 to 1, from highest to worst, and the total is calculated by giving the relative position of each formula with concerning to the quality of the result.

The visual comparison of the samples is made with a white light focused on the side of them to highlight the presence of residues or devitrification on the glass surface.

3.2.2. Optical properties – Transmittance spectra

This technique will be used to compare the transmitted light in the different samples. Comparing the results with those obtained visually. The UV/Vis absorbance spectra were recorded in three different points for each sample using a Varian Cary-5000 UV/VIS/NIR spectrophotometer over the 300-800 nm wavelength range with a 1 nm resolution. As the samples have different thicknesses, the result was divided by the thickness at each point. The range to be subsequently analyzed will be the range of visible light between 400-700 nm. Figure 21

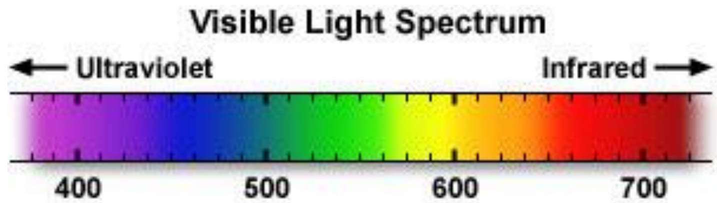

Figure 21: Visible Light Spectrum[55]

3.3. Characterizing the molds.

3.3.1. Analysis of the Mold Surface. Optical Microscopy

To investigate if there is any visible difference in the surface in contact with the glass of the mold, after the firing its finished, images of the mold surface will be taken using a Leica MZ16 stereomicroscope (7.1 × to 115 × zoom range), equipped with a Leica ICD digital camera, and a fiber-optic light Leica system (Leica KI 1500 LCD). The objective is to understand if there are differences in the surface of the mold that can explain the difference in transparency between the samples.

3.3.2. Thermal properties.

To better understand the temperature behavior of the studied molds, their thermal properties will be measured by using the dilatometry technique. The coefficient of thermal expansion is probably the factor that exerts the greatest influence on resistance to thermal shock. The lower the coefficient of expansion, the greater the resistance to thermal shock.[56] In order to analyze if there is a difference in the behavior with the temperature of the molds with the different additives, a Netzsch Dil402PC dilatometer will be used from 25 to 900 °C with a heating rate of 5 K·min^{-} Samples of the four selected formulas will be taken, preheated to 400ºC to eliminate physical and chemical water, to be possible to perform the analysis. Then the samples will be shaped, in the form of cylinders of 2.5 cm length and with a diameter of approximately 0.5 cm.

3.3.3. Do the additives modify the crystalline structure of the molds?

Using an A Rigaku X-ray Diffractometer, model MiniFlex II, with Cu X-ray tube (30 kV/15 mA) was used over a 2θ range of 10–70° with a scanning speed of 2°/min., the crystalline system of the powder samples will be analyzed to analyze whether the addition of these components generates a change in the internal structure of the crystal. X-ray diffraction is a powerful nondestructive technique for characterizing crystalline materials.[57]

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55 https://micro.magnet.fsu.edu/primer/lightandcolor/lightsourcesintro.html
56 1940-, Callister, William D. Jr. *Materials science and engineering : an introduction*. Rethwisch, David G. (8th ed.). Hoboken, NJ. ISBN 9780470419977. OCLC 401168960.
57 https://www.sciencedirect.com/topics/materials-science/x-ray-diffraction

3.4. Understanding other variables in the search for transparency.

3.4.1. Does the pre-drying of the mold affect transparency?

As it is said by Angela Thwaites in her book Mold Making for Glass, silica is a common ingredient in molds, it is reactive with glass at casting temperatures, exchanging ions, thus it adheres to glass. This can be decreased by drying and preheating the molds[58]. Pre-firing empty molds reduce the risk of the mixture adhering to the glass surface, and also reduces the probability of devitrification. It also indicates that the molds should be pre-fired at a temperature a little lower than 100 ºC to prevent the steam from generating cracks in the mold[59].

In order to analyze the effect of the previous drying of the mold on the transparency of the glass, the casting will be carried out with molds with and without previous drying. The molds are made with the 50/50 mix, and the drying will be carried out by bringing the mold to 94ºC for 5 hours. this time was determined by experimentation, watching every hour to ensure the lack of water vapor in the atmosphere of the oven.

TABLE 8 PREVIOUS DRYING CYCLE

STEP	TIME (HRS)	TEMP (ºC)
0	0	24
1	3	94
2	5	94
3	turn off	24

3.4.2. Does consolidation of the mold surface using Shellac improve transparency?

Shellac is a natural material used as a stabilizer and separator. Amalric Walter and other pate-de-Verre artists use it for this purpose[60]. In this experiment we are going to discover if the use of shellac affects the transparency of the final pieces. For which molds will be made using mixture 1 (50% plaster / 50% silica), they will be left to dry for a day, and a layer of shellac will be placed on it.

[58] Mould making for glass, Angela Thwaites. A&C Blaxck, London. Pag27
[59] Mould making for glass, Angela Thwaites. A&C Blaxck, London. Pag88

[60] Keith Cummings. (2009). Contemporary Kiln-formed glass p37

4

Chapter 4. Results analysis.

4.1. Characterization of the glasses.

4.1.1. Calculation of the composition of the glasses

Table 8 shows the composition of the most common glasses, information that will be used to define the type of glass used. Table 9 shows the main function of oxides that were found on the examples and. Table 10, shows the oxides concentrations resultant from the experiment for each glass obtained by XRF analysis. It can be observed that the used glasses are mixed alkaline glasses, in the case of the Turkish and the furnace glass, and the float glass a soda-lime silicate. The oxides highlighted in yellow are the forming oxides, in orange the fluxes and in blue the stabilizers.

TABLE 9 CRITERIA USED TO DISTINGUISH AMONG DIFFERENT TYPES OF GLASS (Cílovà & Woitsch, 2012; Dungworth et al., 2006; Mortimer, 1995; Schalm et al., 2007).

GLASS TYPE	CRITERIA
Soda-rich glass	Na_2O > 10 wt%
Potassium-rich glass	K_2O > 10 wt%
HLLA	CaO > 20 wt%
Mixed alkali glass	Na_2O K_2O in similar concentrations
Lead glass	PbO > 20 wt%

TABLE 10 PROPERTIES OF OXIDES[61]

CLASSIFICATION	OXIDES AND PROPERTIES
FORMERS	SiO_2 Al2O3, Conditional formes
STABILIZERS	ZnO increases resistance to thermal shock [62] Al_2O_3 improves resistance, avoids devitrification, TiO_2 opacifaying, CaO prevents solubility but generates tendency to devitrification, MgO avoid devitrification[58]
FLUXES	K_2O. Generates more shine. Na_2O the higher the quantity, the less viscosity and the greater the solubility. PbO, disol refractorie and Impurity particles, BaO Increases the refractive index SrO,
REFINING	Sb_2O_5
COLORS	Fe_2O_3 , S, TiO_2, Cr_2O_3

TABLE 11 CONCENTRATION OF OXIDES IN THE ANALYZED GLASSES

OXIDE	TURKISH	FURNACE	FLOAT
SiO_2	72,0%	70,00%	71,1%
Na_2O + MgO	10.2%	12.8%	18.7%
K_2O	9,1%	7,6%	0,4%
CaO	4,3%	3,6%	8,2%
BaO	2,2%	4,8%	
CuO	0,5%		
Al_2O_3	0,5%	1,1%	1,2%
Sb2O3	0,3%	0,6%	0,2%
ZnO	0,06%	0,2%	
Cr_2O_3	0,04%		
Cl	0,03%	0,02%	0,01%
SrO	0,03%	0,04%	
PbO	0,03%	0,05%	
TiO2			0,01%
S		0,04%	0,04%
Fe2O3		0,01%	0,10%

FLUXES	**21,6%**	**25.2%**	**19,1%**
STABILIZERS	**4,80%**	**4,7%**	**9,4%**

[61] El vidrio. Jose Maria Fernandez Navarro. Third edition. Artegraf, S.A, p81.

Materias Primas para la Industria del Vidrio. Jesús Ma. Rincón Grupo/Lab de Materiales Vítreos y Cerámicos Instituto E. Torroja de Ciencias de la Construcción, CSrc

[62] Características de las materias primas para la fusión de diferentes tipos de vidrios, J. M? FERNANDEZ NAVARRO Instituto de Cerámica y Vidrio, C.S.I.C. Arganda del Rey (Madrid). P 450-460

The **Turkish glass** has the highest brightness compared with the rest, due to the presence of the highest percentage of K_2O (9,09%). It has the lowest concentration of Al_2O_3 among the three glasses, and does not present MgO (This was possible to determine since the original formula of the manufacturers is available, see annex 6) , so it would has a tendency to devitrify**.**

The **Furnace glass** has the highest refractive index, among then, due to the presence of the highest percentage of BaO (4,77%), it presents a fluidity similar to Turkish glass as it has similar concentrations of fluxes and stabilizers. It has less tendency to devitrify due to the greater presence of Al_2O_3 and less presence of Cao. Given the limitations of the equipment that does not quantifies low weight elements, as MgO and also Na_2O, It is not possible identifying the percentage of MgO, so no more information can be given about the tendency to devitrify.

The **Float glass** is the one with a greater tendency to devitrify as is the one with a higher CaO content, being the least fluid of all. It also has in one side a tin film (due to the float glass manufacture) that may facilitate the devitrification.

4.1.2. Thermal Properties.

The result of the characterization of the glass can be seen in table 14. The calculation of the coefficient allows us to analyze which glass has greater resistance to thermal shock, as well as whether there is a correlation between the Tg, and COE (α) with the degree of devitrification of the glasses. It would be expected that within the same formulation Turkish glass would have the greatest devitrification, having the lower Tg, and Ts, so at the same casting temperature with respect to the other glasses followed by furnace and finally float.

TABLE 12 THERMAL PROPERTIES OF THE DIFFERENT GLASSES

Glass	Glass Transition Temperature (Tg) / ºC	Softening Temperatures (Ts) ºC	Coefficient of Expansion (α)
Float	542.5	597.2	7.84×10^{-06}
Furnace	487.0	531,5	11.11×10^{-06}
Turco	471.6	520.8	12.42×10^{-06}

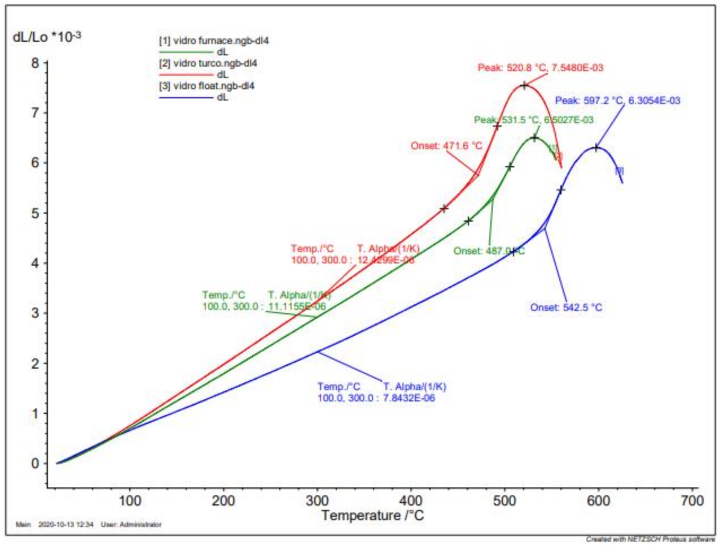


Figure 22 Dilatometry curve with the thermal behavior of the different analyzed glasses, between 0 - 850ºC

4.2. Analysis of the Glass casting samples

4.2.1. Correlation between thermal properties and devitrification

As was mentioned above, the objective of this experiment is to determine if there is a correlation between the Tg, and COE with the degree of devitrification of the glasses. It would be expected that those glasses with a higher transformation temperature would present a lower degree of devitrification since it would be less reactive with the walls of the mold. The composition of the glass must be taken into account, so the result may be more accurate when analyzing Turkish glass versus furnace glass, as they have similar compositions. Since the Turkish glass has lower Ts and Tg, it is expected that this glass would has the higher level of devitrification and the float glass the lower.

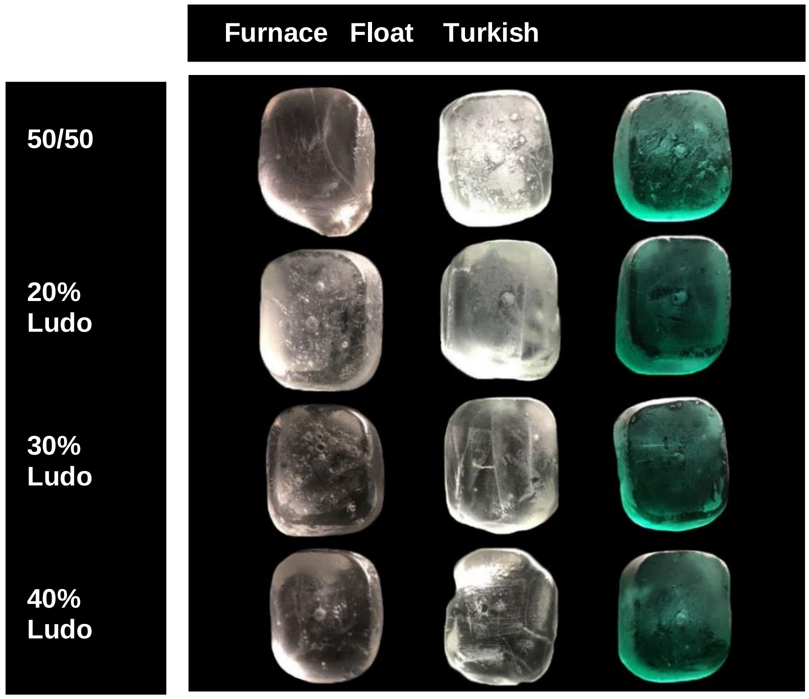


Figure 23 Samples 1 vs 2-4 showing the different degree of devitrification.

It is observed that all the pieces of the Turkish glass present a greater devitrification compared with furnace glass

The Furnace glass has a similar composition when compared to Turkish glass in terms of the proportion of fluxes and stabilizers, however, it has a higher Tg than Turkish glass. The observed difference can be one of the reasons why this glass would suffer less devitrification under the same conditions.

.It was expected that float glass having the highest Tg and COE, would have had less devitrification that the other glass, In this case, float glass does not behave as expected, but as we found in its compositions, this glass has a more tendency to devitrify that the others and also it has a layer of tin in its surface so this results can not be conclusive. The results seem to indicate that between two glasses of similar composition, the Tg could indicate which of them could present greater devitrification, the higher the tg, the lower the devitrification and vice versa, which would help to understand the maximum casting temperature to be used. The analysis was carried out with only two glasses of similar composition, therefore valid conclusions cannot be generated, so a more detailed study of this topic can be carried out.

4.2.2. Moulds formulation

4.2.2.1. Sorting the samples from best to worst results by type of glass

The results of the *firing* cycles of the formulations selected in point 3.2.1 are shown below. After obtaining the samples, a visual comparison was made between them, taking Sample 1 (50/50) as a basis for comparison. Sorting them from highest to lowest quality, considering quality as the absence of devitrification or residues adhered to the glass, and only using a visual analysis. Results for smooth samples are shown here. The calculations and textured samples can be seen in annex No. 1, p.59. The yellow lines indicate the best sample of each formulation.

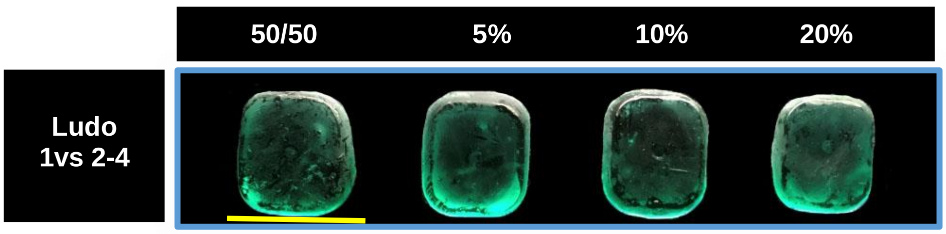


Figure 24 Comparison of Non textured Turkish glass samples. From formulas using Ludo.

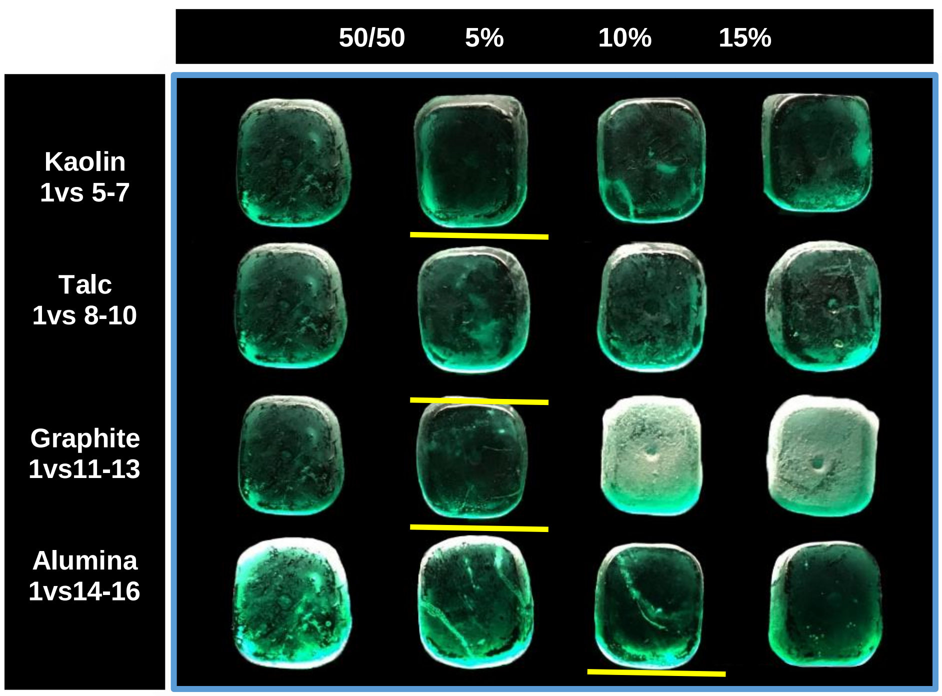


Figure 25 Comparison of Non textured Turkish glass samples. Those marked with the yellow line represent the best option within each category.

TABLE 13 VISUAL CLASSIFICATION OF TEXTURED TURKISH GLASS

GLASS	EXAMPLES	BEST			WORST
		1st	2nd	3th	4th
TURKISH GLASS WITHOUT TEXTURE	**Ludo: 1 vs 2-4.**	**1**	**3**	**4**	**2**
	Kaolin: 1 vs 5-7,	**5**	**6**	**7**	**1**
	Talc: 1 vs 8-10.	**8**	**1**	**9**	**10**
	Graphite: 1 vs 11-13.	**11**	**1**	**12**	**13**
	Alumina: 1 vs 14-16	**15**	**14**	**1**	**16**

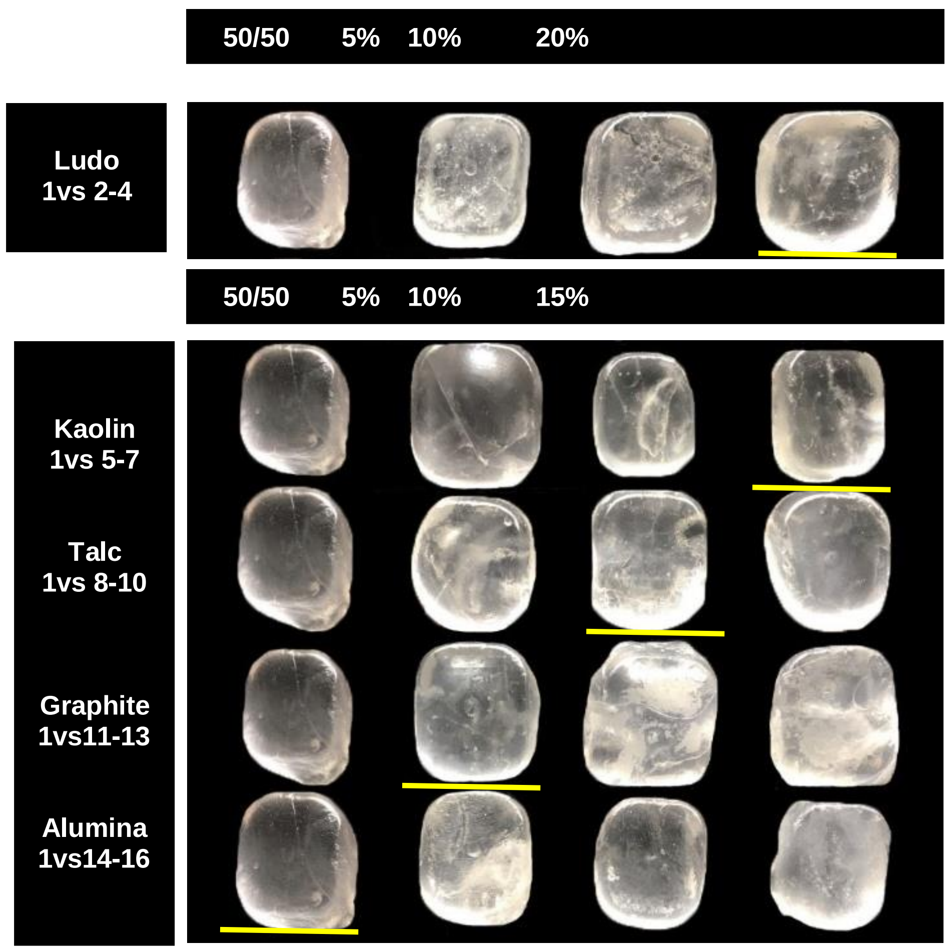


Figure 26 Comparison of Non textured Furnace glass samples. Those marked with the yellow line represent the best option within each category

TABLE 14 VISUAL CLASSIFICATION OF NON TEXTURED FURNACE GLASS

GLASS	EXAMPLES	BEST 1ST	WORST 2ND	3TH	4TH
FURNACE GLASS WITHOUT TEXTURE	**Ludo: 1 vs 2-4.**	4	3	1	2
	Kaolin: 1 vs 5-7,	7	5	6	1
	Talc: 1 vs 8-10.	9	1	8	10
	Graphite: 1 vs 11-13.	11	12	1	13
	Alumina: 1 vs 14-16	1	14	15	16

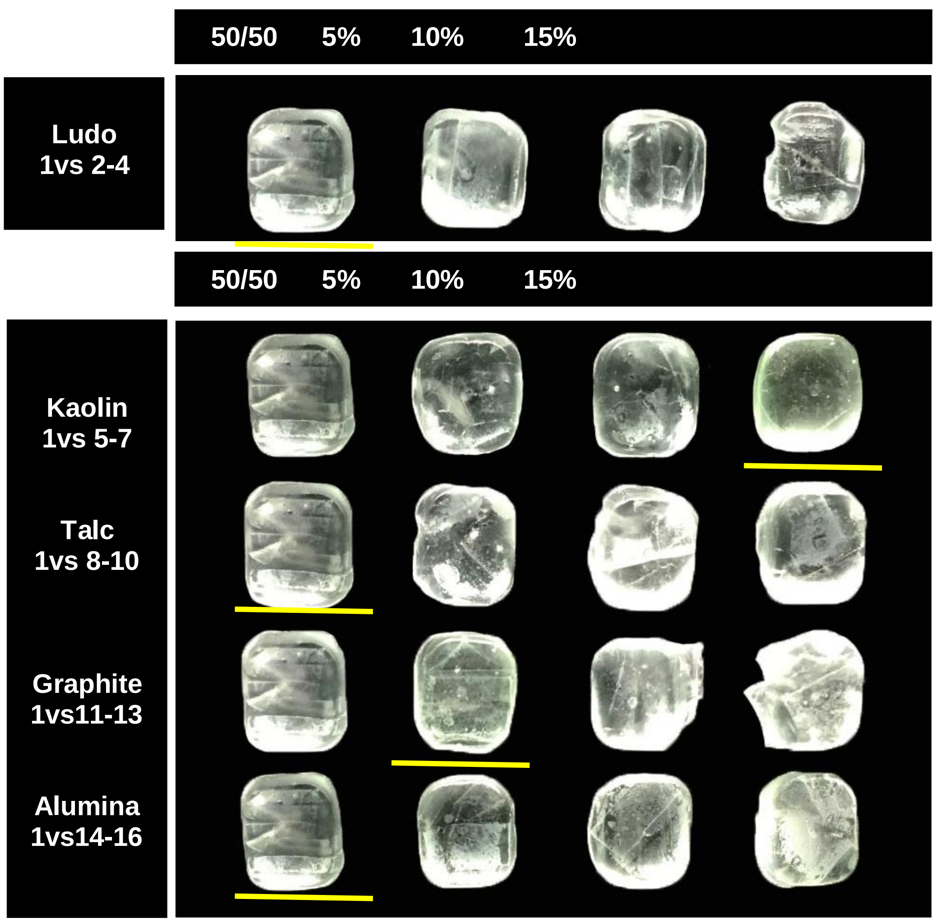


Figure 27 Comparison of Non textured Float glass samples. Those marked with the yellow line represent the best option within each category

TABLE 15 VISUAL CLASSIFICATION OF NON TEXTURED WINDOW GLASS

4.2.2.2. Selection of the best formulas

GLASS	EXAMPLES	BEST		WORST	
		1ST	2ND	3TH	4TH
WINDOW GLASS WITHOUT TEXTURE	**Ludo: 1 vs 2-4.**	**1**	**4**	**3**	**2**
	Kaolin: 1 vs 5-7,	**7**	**5**	**6**	**1**
	Talc: 1 vs 8-10.	**1**	**8**	**10**	**9**
	Graphite: 1 vs 11-13.	**11**	**1**	**12**	**13**
	Alumina: 1 vs 14-16	**1**	**14**	**15**	**16**

In order to obtain the best 4 mold formulas, a visual order was made between them from best (less devitrification and glued residues) to worse (greater devitrification and glued residues) following the sequence of analysis. The calculations can be seen in annex No. 2.

TABLE 16 RELATIVE POSITION OF SAMPLES

ADDITIVE	BEST			WORST
1 vs Ludo	**4** (20%Ludo)	**1**	**3**	**2**
1 vs Kaolin	**7** (15% Kaolin)	**5**	**6**	**1**
1 vs Talc	**1** (50/50)	**8**	**9**	**10**
1 vs Graphite	**11** (5 %Graphite)	**1**	**12**	**13**
1 vs Alumina	**1** (50/50)	**14**	**15**	**16**

The mold formulas that present better visual results are numbers 1, 4, 7 and 11, and it will be with these formulas, that the rest of the experimentation will continue.

TABLE 17 RELATIVE POSITION OF THE BEST SAMPLES

		BEST		WORST		
		1st	**2nd**	**3th**	**4th**	**total**
Examples	**Points**	4	3	2	1	
1	**Frequency**	0	0	3	3	9
4		0	1	2	3	10
7		3	2	1	0	20
11		3	3	0	0	21
		11	**7**	**4**	**1**	

According to the results obtained, the formula that generates greater transparency is number 11 with 5% Graphite, followed by the use of 15% Kaolin, 20% Ludo and finely the 50/50 base formula.

The Results of grouping and sorting the samples are:

The best sample using **Ludo** is number 4 (20% Ludo). To use of Ludo improves the transparency but not significantly, therefore, its use can be recommended to reduce the cost of materials in the molds. Given that this is one of the 4 better formulas, and given the impact, it would have on reducing costs. The analysis will be carried out by increasing up to 50% of the formulation, this is the maximum percentage recommended in the literature

The use of **Kaolin** consistently improved the transparency of the pieces, the higher the proportion, the greater the transparency. The best option is through Sample 7, using Kaolin at 15%

The use of **Talc** is not conclusive. It does not seem to generate an important improvement in the transparency of the samples.

The best formula using Graphite is number 11 (5% Graphite). Graphite improves transparency depending on the proportion used. The higher the proportion of Graphite, the samples show greater devitrification, the use of Graphite in large proportions generates a higher devitrification, presenting the glass an aged appearance, which can be used as a decorative aspect in the casting pieces. An additional study will be carried out with this element, reducing its composition between 5-1%.

The use of Alumina mixed with silica, in the proportions used, does not show to give better results than using only silica, in this case it's better to use the 50/50 formula. Additional studies can be done by increasing the proportion of Alumina.

4.2.2.3. Decreasing the proportions of Graphite

Given that the use of Graphite was found to generate good transparency in the glass, this analysis is intended to find the minimum amount of the element that can be used to obtain good results. The percentage of Graphite will vary from 5% to 0.5%. These results will be joined with those previously obtained with proportions ranging from 5 % to 15% (see figures 24-26), and a visual comparison of the samples will be carried out, to finally make a ranking of the samples from best to worst. The results can be seen in table 18, where it is tabulated from 1 to 6, to obtain quantitatively those samples that present better transparency with 1 being the result with less transparency, and 6 the one with the greatest transparency.

TABLE 18 ORDERING OF THE RESULTS FROM BEST (6) TO WORST (1) ACCORDING TO THE TRANSPARENCY OF THE SAMPLES

GLASS	GRAPHITE					
	5%	**2%**	**1%**	**0.5%**	**15%**	**10%**
FURNACE	6	5	4	3	1	2
TURKISH	6	5	4	3	1	2
FLOAT	6	5	4	3	1	2
AVERAGE	**18**	**15**	**12**	**9**	**3**	**6**

The results are conclusive, the concentration of 5% is the one that presents the best result (see figure 34). Higher concentrations cause an accumulation of the material on the surface generating a great devitrification, this result could be used artistically to give the glass a stone look. At lower concentration, the result shows less transparency.

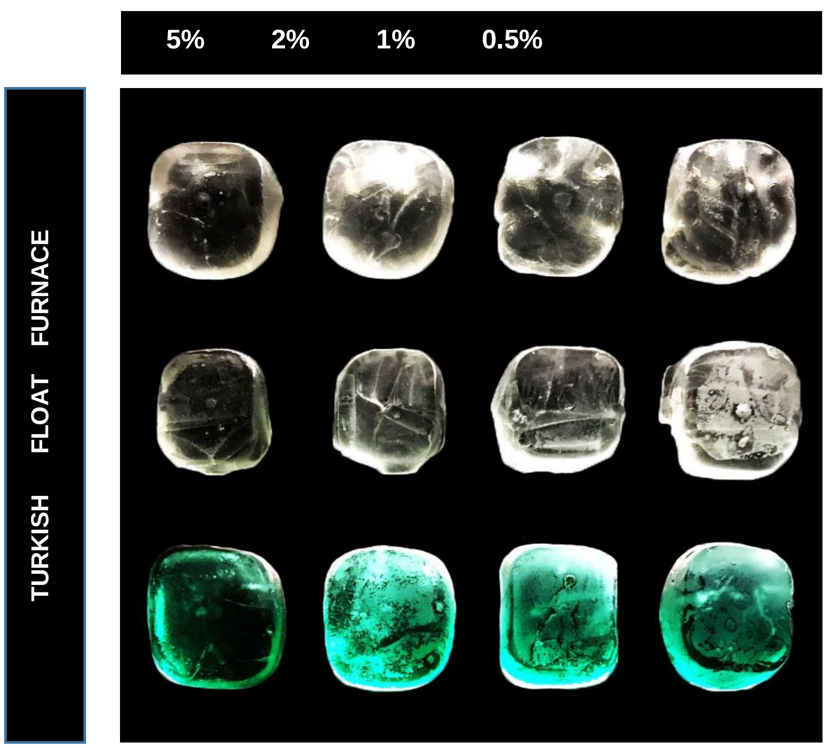


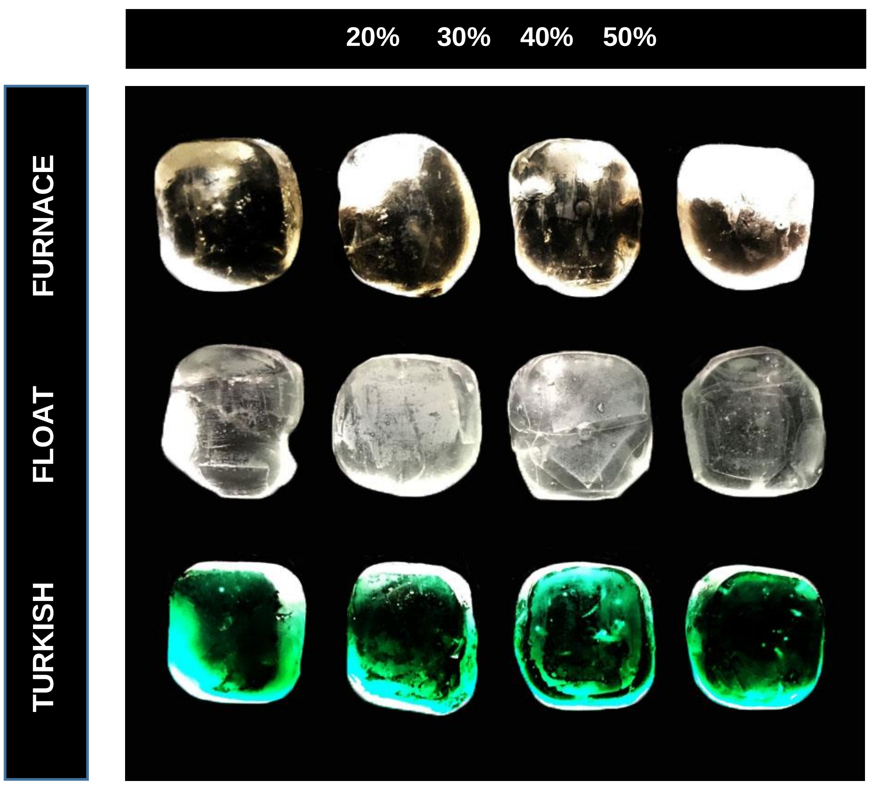
Figure 28 Samples resulting from using lower concentrations of Graphite

4.2.2.4. Increasing the proportions of Ludo

Given that the use of Ludo was found to generate a little bit more transparency than then base formula, and given the impact it would have on reducing costs, it is decided to do this analysis were the Ludo proportion will be increasing up to 50% of the formulation, this is the maximum percentage recommended in the literature[63]. The effect of different concentrations of Ludo in the samples was analyzed, from 20% to 50%, the results can be seen in table 19 where it is ranked from 1 to 4 with 1 being the result with less transparency, and 4 the one with the greatest transparency.

TABLE 19 ORDERING OF THE RESULTS FROM BEST (4) TO WORST (1) ACCORDING TO THE TRANSPARENCY OF THE SAMPLES

Glass	20% Ludo	30% Ludo	40% Ludo	50% Ludo
Furnace	4	2	1	3
Turkish	1	3	2	4
Float	3	1	2	4
AVERAGE	**8**	**6**	**5**	**11**



63 Angela Thwaites. (2011). Mould Making for glass

Figure 29 Samples resulting from using lower concentrations of Graphite

The use of Ludo slightly improves the transparency of the pieces when compared with the base formula (50% plaster / 50% silica), but it is not possible to say with these results if a higher concentration of Ludo generates more transparency since in the experiment the highest transparency was achieved at the extremes, using 50% and 20%. Ludo can be used to reduce the costs of the mold. Its use generates a shorter setting time of the Plaster, so it must be quickly emptied on the positive or increase the proportion of water.

4.2.3. Transmittance Spectra.

Visually glass samples made in the molds with the formulations 11 (5% Graphite), 7 (15% Kaolin), 4 (20% Ludo and 1 (50%plaster / 50% silica) were the ones with better results concerning transparency, therefore these glass and molds samples were analyzed in more detailed. Below we can observe the spectra representing the transmittance of the previously considered samples. The range of visible light was considered between 380 and 750 nm. The furnace glass and the float, as non-colored glass samples, transmit light in all the visible range, however, the spectra of the Turkish glass present a broad band with a maximum around 750 nm, which corresponds to the absorption of Cu^{2+}. [64]The presence of copper in these samples was also observed in the XRF analysis. For the furnace glass, the highest transmittance is given by Graphite and Kaolin, followed by the 50/50 formula, and finally with a relative difference of approximately 10% the formula used by Ludo.

[64] El vidrio. Jose Maria Fernandez Navarro. Third edition. Artegraf, S.A.

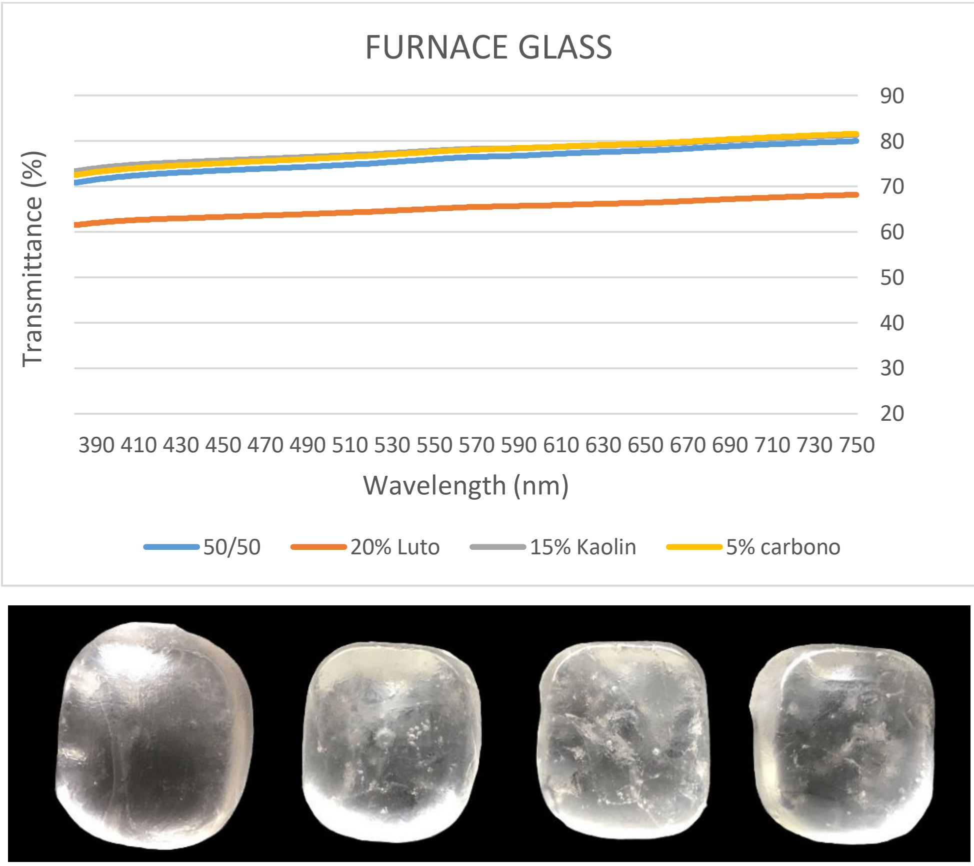
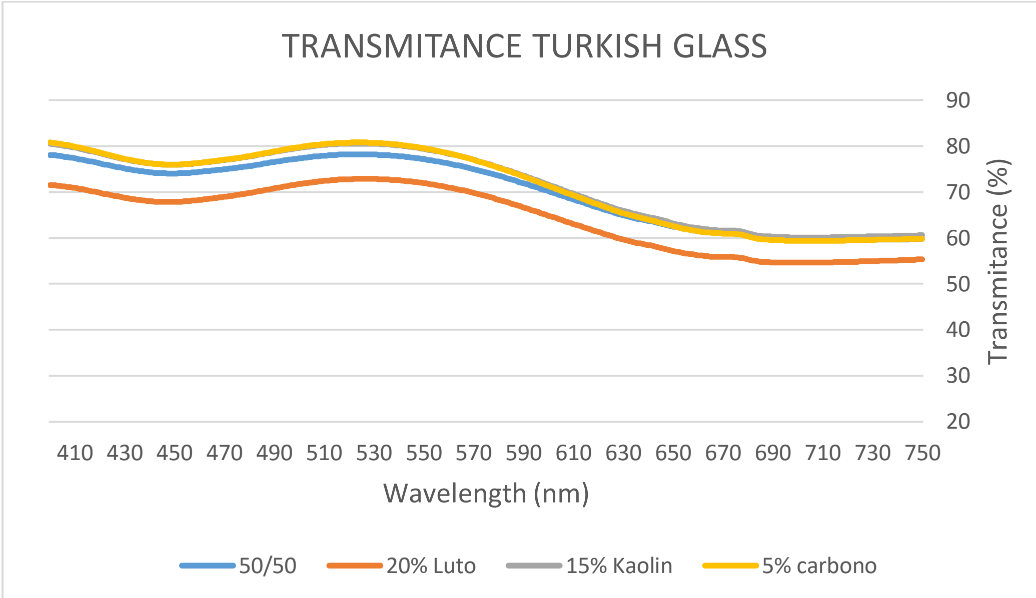


Figure 30 Transmittance spectra and a photograph with the furnace glass samples. From right to left samples 1- 4-7-11

For the Turkish glass, the highest transmittance is given by Graphite and Kaolin, followed by the 50/50 formula, and finally with a relative difference of approximately 5 % the formula used by Ludo.



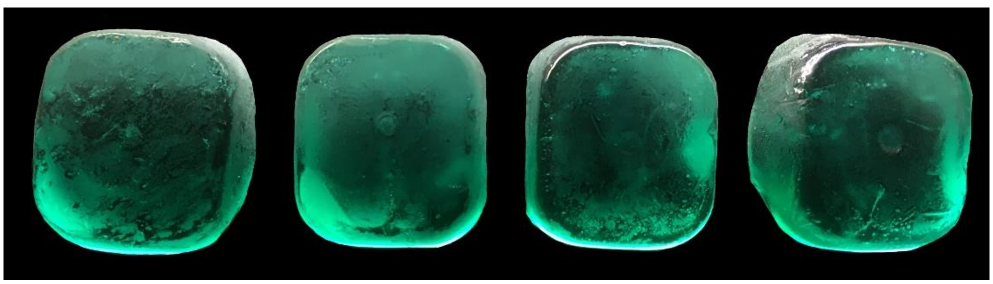


Figure 31 Transmittance spectra and photograph of Turkish glass. From right to left samples 1- 4- 7-11

For the float glass, the highest transmittance is given by Kaolin followed by Graphite. In the float case the formula 4 it's better than 50/50.

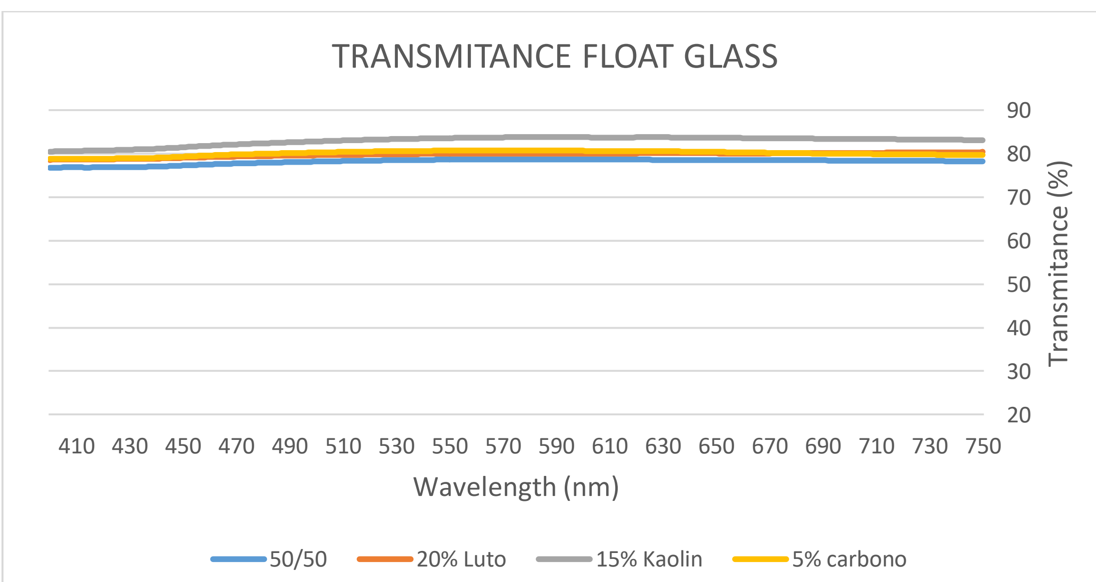


Figure 32 Transmittance spectra and photograph of Float glass. From right to left samples 1- 4- 7-11

4.2.4. Homogeneity Analysis

As can be seen in the previous images, the samples are not homogeneous on their surface, presenting areas of greater and lesser transparency, so it was decided to calculate how homogeneous the pieces are by calculating the standard deviation of their results. Three different spectrum was made in the same sample in three different areas, then the analysis is achieved by calculating the standard deviation of the average value of transmittance, between 350 and 750nm. The standard deviation of the different samples is compared and the one with the lowest value is selected. The higher the number, the greater the range of variation and therefore less homogeneity.

TABLE 20 EXAMPLE OF THE CALCULATION OF STANDAR DEVIATION OF THE TRANSMITANCE IN THE SAMPLES FOR FURNACE GLASS

Average trasmittance Point a	Average transmittance Point b	Average trasmittance Point c
77,66	78,13	71,04
STANDARD DEVIATION: 3.96		

TABLE 21 SURFACE HOMOGENEITY ANALYSIS, STANDAR DEVIATION OF THE TRANSMITANCE

GLASS	50/50	20% LUTO	15% KAOLIN	5% GRAPHITE
FURNACE	3,96	2,85	1,51	1,88
TURKISH	3,36	3,57	2,19	1,56
FLOAT	0,82	4,58	1,93	1,56
AVERAGE	**2,71**	**3,67**	**1,88**	**1,67**

Considering the differences in the transmittance spectra obtained for the same sample (Table 21), it can be observed that the use of Graphite at 5% generates greater homogeneity in the samples followed by Kaolin. The least homogeneity is given by the use of Ludo (see annex 4). This is in agreement with what is seen in the glass samples.

In the case of Furnace glass, the best result is by using Kaolin or Graphite. The difference in transmittance between the first 3 is not representative, but if we consider the homogeneity of the samples, shown in table 21, the best option in this case is using Kaolin.

In the case of Turkish glass the best options are through the use of Kaolin or Graphite, when combined with homogeneity the best option will be using Graphite.

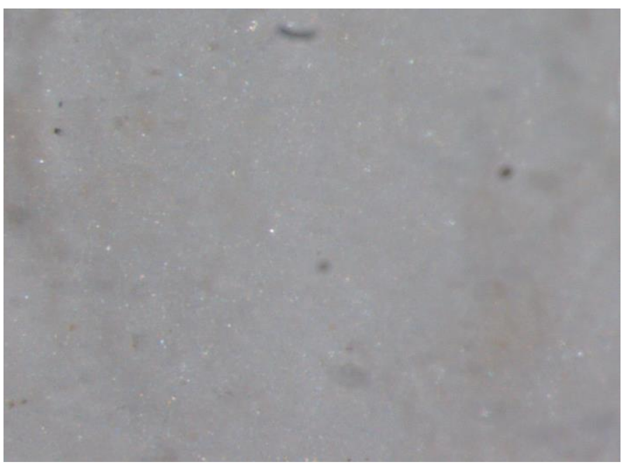
For float glass, the highest transmittance is achieved by the use of Kaolin, although the highest homogeneity is obtained with Graphite.

In all the samples, the one that generates the lowest transmittance is mixture 4 and 1, and they also present the highest range, so they present the least homogeneity.

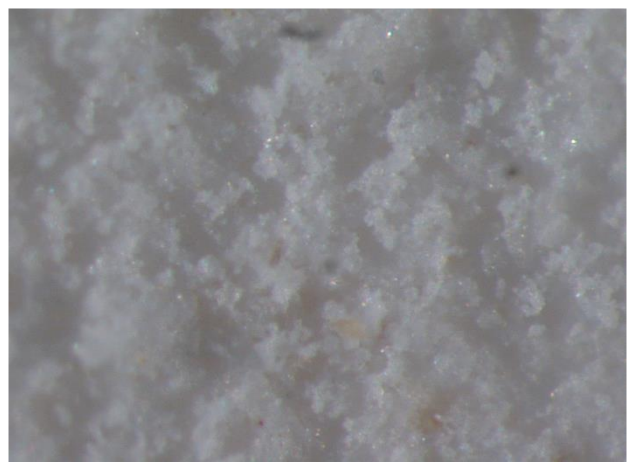
4.3. Characterizing the moulds.

4.3.1. Analysis of the Mold Surface. Optical Microscopy

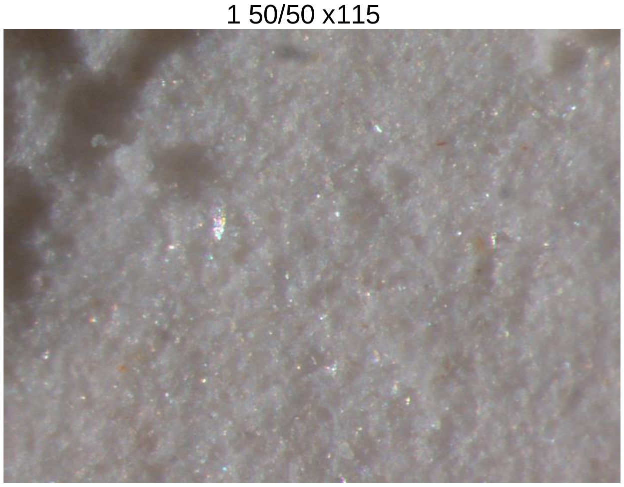
To investigate if there is any visible difference in the surface in contact with the glass of the mold, after the firing, images of the surface were taken The results can be seen in the following images:



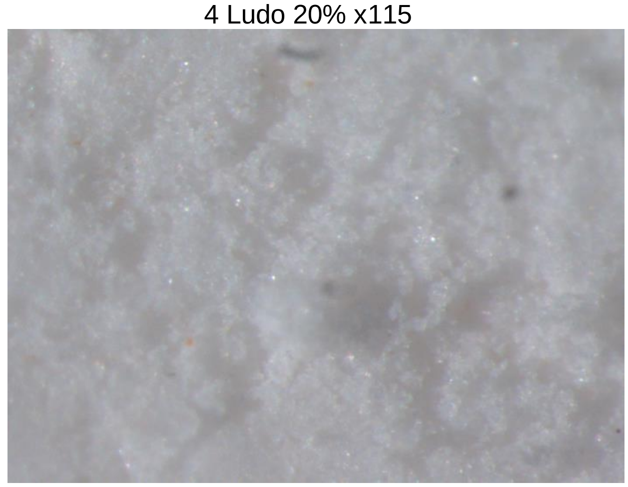
1 50/50 x115



4 Ludo 20% x115



7 Kaolin 15% x115



11 Graphite 5% x115

Figure 33 Optical Microscopy imagen

The surface of mix 1 is the least rough of the 3, presenting a smooth appearance. The difference in the roughness of the samples can be explained by the differences in granulometry of the different additives. The variation of granulometry contributes to give a better packing to the mold, giving greater strength and density to it, which is beneficial to stop the moulds for glass, and also it may be due to traces of the mold that are stuck to the glass.

The sample 4 presents the highest roughness, and this can be explained because the granulometry of Ludo is greater than that of Plaster and Silica.

After seeing the results, it is not possible to get a conclusion since it would be necessary to carry out additional tests, where the surface of the moulds before and after the firing cycle is compared, without containing glass, so in this way it could be possible to know if the appearance shown is due to the granulometry of the investment or rit is due remnants of the investment that were attached to the glass when it was removed, this would give an idea of the strength of the mold.

4.3.2. Thermal properties of the different moulds formulations

With this analysis we seek to understand the behavior of the different formulas with the variation of temperature. The thermal properties were measured by using the dilatometry technique. The coefficient of thermal expansion is probably the factor that exerts the greatest influence on resistance to thermal shock[65] [66]. The lower the coefficient of expansion, the greater the resistance to thermal shock. The results of the experiment can be seen in table 22.

TABLE 22 VALUES OF THE COEFFICIENT OF EXPANSION AND TRANSFORMATION TEMPERATURE OF THE DIFFERENT MOLD FORMULAS

FORMULA	Tg ºC	Linear expansion coefficient (200-400 ºC)	**Δ λ** */ formula base* *%*
v	569.8	$17.30x10^{-06}$	
4 - Ludo 20%	567.3	$15.17x10^{-06}$	35%
7 - Kaolin 15%	562.4	$11.85\ x10^{-06}$	58%
11 - Graphite 5%	561.8	$16.88x10^{-06}$	22%

65 https://micro.magnet.fsu.edu/primer/lightandcolor/lightsourcesintro.html
66 https://en.wikipedia.org/wiki/Thermal_stress

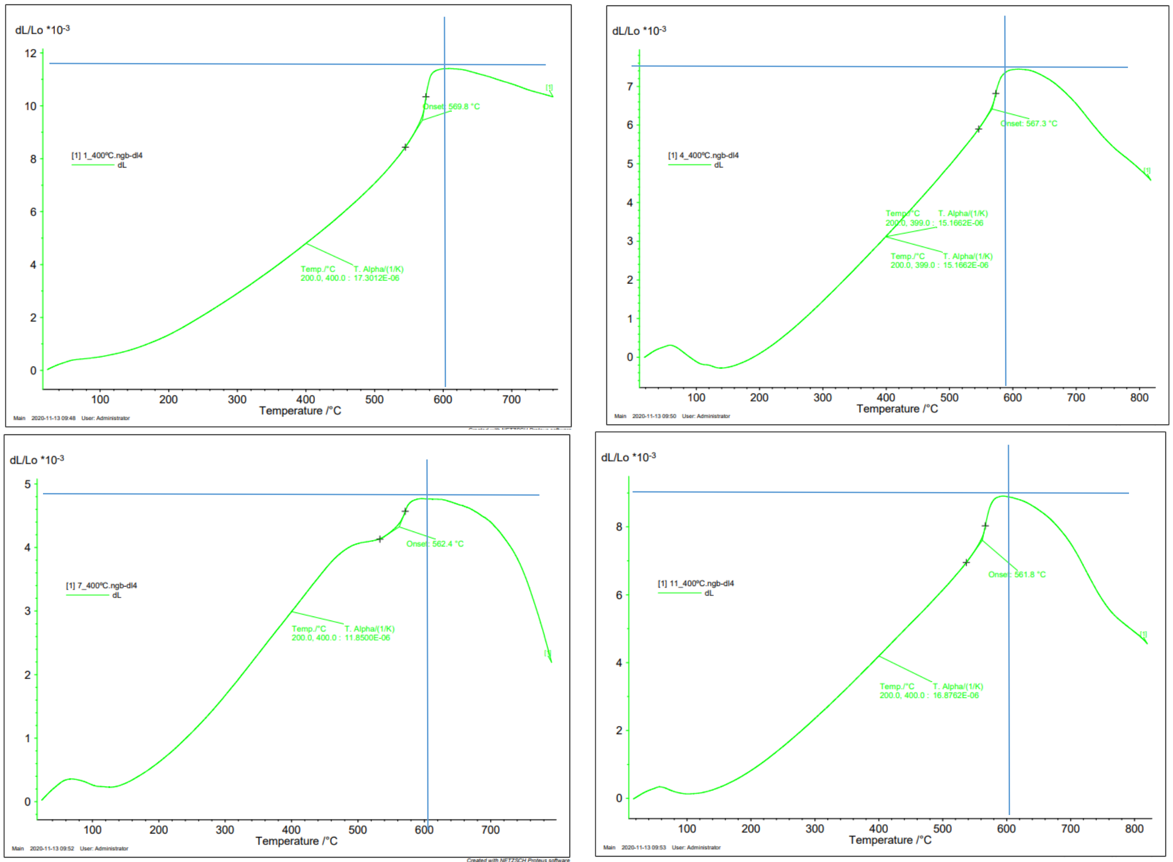


Figure34 Dilatometry curve of the selected formulations

According to the results obtained, the use of Ludo increases the resistance to thermal shock in the moulds by 35% compared to 50/50. Kaolin is the additive that generates the greatest variation in the coefficient of expansion, reducing it by 58%. Likewise, the use of Graphite as an additive reduces the coefficient of expansion by 22%. In summary, the use of these additives improves resistance to thermal shock of moulds compared to base formula 50/50.

4.3.3. Do the additives modify the crystalline structure of the molds?

Samples of the molds were pulverized after being fired up to a temperature of 850 °C, and the characterization of the crystals contained was carried out through the use of XRD. To identify the minerals present in the structure, the peaks of the curves obtained were compared with reference values that identify the minerals of Cristobalite, Quartz and Anhydrite. In order to compare the values with each other, they were normalized based on 100, and the results can be seen in figure 35

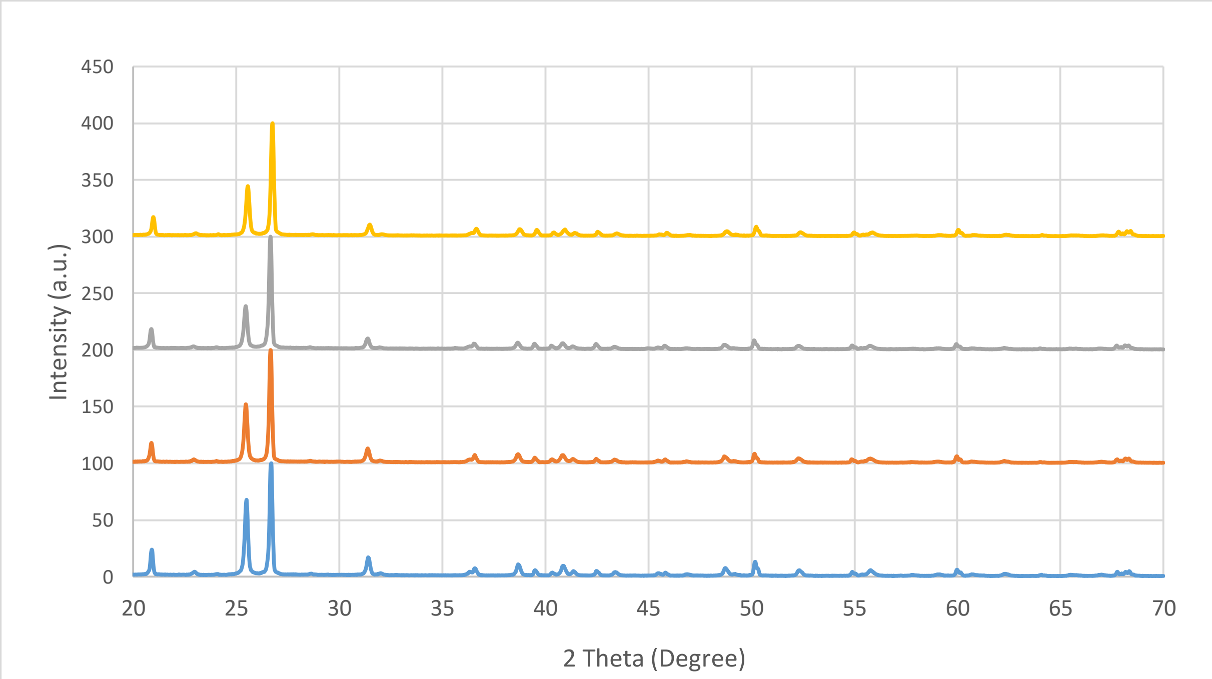


Figure 35 X-ray Powder Diffraction (XRD)

TABLE 23 NORMALIZED PROPORTIONS OF QUARTZ AND ANHYDRITE IN THE SAMPLES

	50/50	20% Ludo	15% caolin	5% grafito
Anhydrite	100.00	100.00	100.00	100.00
Quartz	67.72	52.16	38.72	44.60
Proportion	67.72	52.16	38.72	44.60

The results indicate that the samples that present the greatest transparency (15%, kaolin and 5% graphite) are in turn those that show the least quartz crystals in the structure.

The 50/50 mixture is the one that generates the least transparency, and is in turn the one that shows a greater amount of quartz in the structure.

4.4. Understanding other variables in the search for transparency.

4.4.1. Does the pre-drying of the mold affect the transparency?

In order to analyze the effect of the previous drying of the mold on the transparency of the glass, the casting was carried out with moulds with and without previous drying. The moulds were made with the 50/50 mix, and the drying was carried out by bringing the mold to 94ºC for 5 hours.

In the figure 35, it is possible to see the effect of drying out of the moulds previously to the casting firing. The Dried pieces are the right and without drying on the left.

Figure 36 Samples of glass with drying moulds on the right and without drying moulds on the left.

As it can be seen in the three types of glass, the previous drying of the moulds at 94 ºc generates better transparency, in the glass. It is then verified that for all types of glass the pre-drying of the mold improves the transparency of the resulting pieces

4.4.2. Does consolidation of the mold surface using Shellac improve transparency?

In this experiment we are going to discover if the use of shellac affects the transparency of the final pieces. For which moulds were made using mixture 1 (50% plaster / 50% silica), they were left to dry for a day, and a layer of shellac was placed on it. In the figure 31, it is possible to see the effect of using shellac in the moulds previously to the casting firing. A special effect on the transparency of the glass is not visualized by the use of Shellac, although it works very well to stabilize the mixture avoiding that Plaster particles are trapped in the glass.

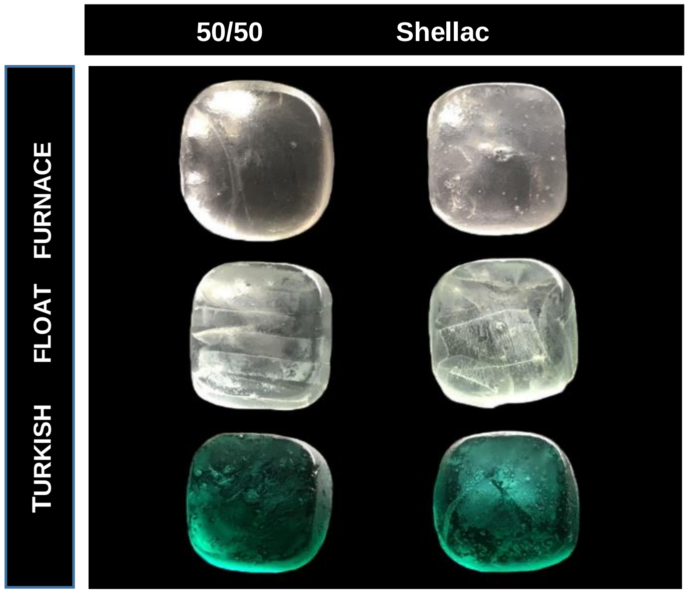


Figure 37 Samples resulting from using Shellac

5

5. Chapter 5. Cost Analysis and Conclusions

5.1 Cost Analysis

The present analysis is carried out to compare the costs of the proposed formulas versus the cost per kilogram of the marketed product. In table 25 it can be seen the average costs per kilogram of pre-made material. The cost was obtained by investigating the prices of some brands mentioned in the literature. It can also be seen the cost per kilogram of the material separately. It is found that economically it is more convenient to generate any of the recommended formulas using Kaolin or Graphite, than to buy pre-mixed formulas. Using Ludo can generate up to 50% additional cost reduction.

TABLE 24 AVERAGE COSTS OF PRE-MIXED MATERIALS IN THE MARKET AND UNMIXED MATERIAL COSTS

AVERAGE VALUES PRE-MIXED MATERIALS	
COST ($)	51.8
WEIGHT (KGR)	20
COST/KGR	3.56
http://shop.ransom-randolph.com/ https://www.statesupply.com/bl1052 www.luminarglass.com http://www.zrci.com/our-materials-2/	

UNMIXED MATERIAL COSTS				
MATERIAL	KGRS	COST EURO	COST / KGR	SOURCE
PLASTER	25	15	0.6	
SILICA	25	21	0.84	
KAOLIN	1	0.4	0.4	https://spanish.alibaba.com/product-detail/Washed-Kaolin-and-calcined-Kaolin-clay-60835801469.html?spm=a2700.galleryofferlist.normal_offer.d_image.25a5511edFMw6a&s=p
GRAPHITE	1	0.6	0.6	https://spanish.alibaba.com/product-detail/Thermal-conductive-Graphite-powder-black-Graphite-60815935080.html?spm=a2700.galleryofferlist.normal_offer.d_title.6e843c8dMqeUPC&s=p

TABLE 25 COSTS PER KGR FOR THE FINAL FORMULAS ANALYZED

Formula	Plaster %	Silica %	Ludo %	Aditive %
50/50	50	50	0	0
15% Kaolin	50	50	0	15
5% Graphite	50	50	0	5
20% Ludo	40	40	20	0
30% Ludo	35	35	30	0
40% Ludo	30	30	40	0
50% Ludo	25	25	50	0

Formula	Plaster %	Silica %	Ludo %	Aditive %	Cost / kgr
50/50	0,5	0,5	0	0	**0,9**
15% Kaolin	0,5	0,5	0	0,15	**0,96**
5% Graphite	0,5	0,5	0	0,05	**0,93**
20% Ludo	0,4	0,4	0,2	0	**0,72**
30% Ludo	0,35	0,35	0,3	0	**0,63**
40% Ludo	0,3	0,3	0,4	0	**0,54**
50% Ludo	0,25	0,25	0,5	0	**0,45**

Conclusions

Different factors can affect the transparency of the casting pieces, and in this work it has been investigated some of them, factors such as the components that make up the investment of the mold, the drying process of it, and the formulation of the glass, factors that help to obtain a clean and more transparent surface.

One of the important aspects to take into account when looking for transparency in casting pieces is the type of glass used. Depending on its composition, the working temperature can vary in a range of 787º - 850º. The analysis of the component oxides of the glass allows us to identify if it has a tendency to devitrify or not, as well as the knowledge of softening point Tg and the coefficient Of expansion COE, gives us an idea of its thermal behavior. This aspect is important because the results suggest that depending on the Tg value, the working temperature could be adjusted to achieve the adequate viscosity and thus generate less adherence of the glass to the mold walls. In this thesis, the working temperature was defined at 850 ºC as a fixed variable, it would remain in future research to analyze how depending on the Tg of the glass the maximum working temperature can be modified.

Another point analyzed was the influence of the components of the mold with the level of transparency of the glass. We worked with 5 additives added in different proportions to the base mixture of plaster + silica, the additives were Kaolin, Graphite, Talc Alumina and Ludo.

It was found that the use of Kaolin (chemically inert so will not react with the glass surface), it is useful to release the glass from the mold and improve the transparency and homogeneity of the pieces in casting, in the 3 types of glass analyzed. It was experimented with concentrations between 5% and 15%, resulting that the higher the concentration, the greater the transparency. Using Kaolin also generates variation in the coefficient of expansion, reducing it by 58%, when it is compared to the base formula which implies that this component improves the resistance to thermal shock in the mold.

Despite not being experimentally proven, experience when handling already fired moulds seems to indicate that the material is more fragile after been fired, making it easier to remove from the glass. In cases of large moulds or that are designed to contain large volumes of glass, it would be advisable to use the Kaolin in the first layer, which will be in contact with the glass and then place a 50/50 mixture with Ludo as a reinforcement. A possible Investing formula can be:

For the first layer: 35% Plaster + 30% Silica + 20% Ludo + 15% Kaolin,

For the Backup layer: 40% Plaster + 40% Silica + 20% Ludo

With respect to Graphite, it was found that its use improves transparency but depending on the concentration that it is used. A concentration of 5% of Graphite is the one that presents the best result, both in transparency and in homogeneity, in all types of glass analyzed. Higher concentrations cause an accumulation of the material on the surface generating a great devitrification, this result could be used artistically to give the glass a stone look. A lower percentage in the use of Graphite also reduces the transparency obtained. Another found is that with Graphite at 5% reduces the expansion coefficient by 22% when compared to the base formula, which implies that this component improves the resistance to thermal shock in the mold.

A possible Investing formula can be: 37, 5% Plaster + 37, 5% Silica + 20% Ludo + 5% Graphite

It is advisable to mix all the components dry first before placing the mixture in the water, since the Graphite when placed directly in the water generates a film that makes it difficult to subsequently add the plaster plus the silica.

A comparison of the use of kaolin and graphite as an additive can be seen in figures 38 and 39 where the effect of the use of the additive in the upper part of the samples can be seen.

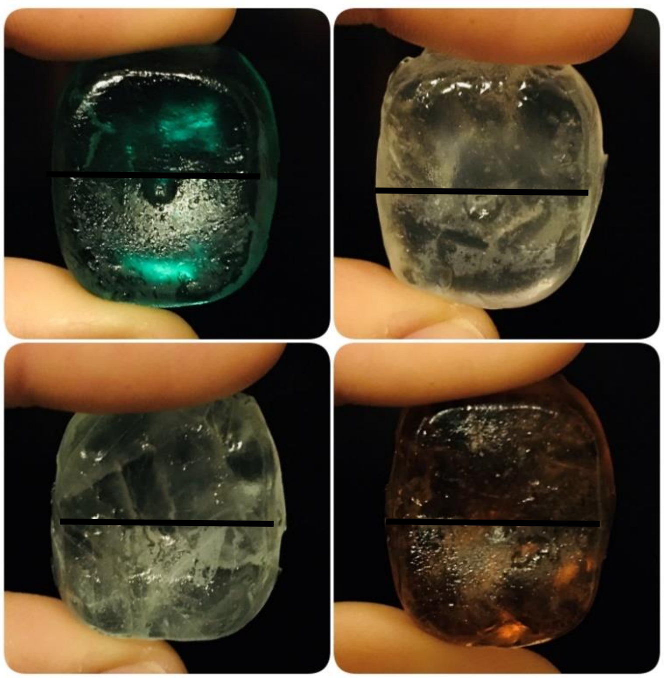


Figure 38 Transparency comparison using Kaolin

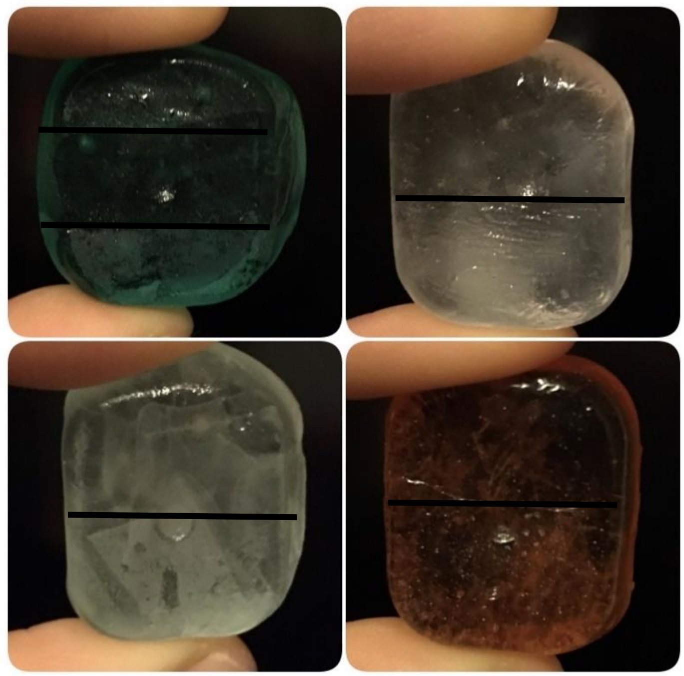


Figure 39 Transparency comparison using Graphite

Regarding Ludo (pre-baked material), the impact of using it in the mold, and the transparency of the pieces, were analyzed. The use of Ludo in between 20% to 50%, improves the transparency when it is compared with the base formula (50/50) but not as significantly as the Kaolin or the Graphite. Therefore, its use can be recommended to reduce the cost of materials in the moulds, the use of Ludo in the formula generates a cost reduction between 20 and 50% depending on the percentage used. Additionally, it follows that it must reduce the cost of drying the mold by reducing the presence of chemical water in it. This aspect was not scientifically proven in this thesis. The use of Ludo decreases the coefficient of expansion of the material and therefore on the increase the resistance to thermal shock of the mold. In the experiment the COE was reduced in 35%. Using Ludo generates a shorter setting time, therefore it is recommended to increase the proportion of water or it must be poured more quickly.

The use of Alumina does not improve the transparency of the pieces, in the proportion used, between 5% - 15%, so in this proportions seems to be better to use the 50/50 formula. It is possible to continue investigating this material by increasing the proportions, since the Alumina being a non-reactive element with glass, it is assumed that it can generate better results by increasing its proportion using it as a refractory material rather than as an additive.

The use of Talc is not conclusive. It does not seem to generate an important improvement in the transparency of the samples. Therefore, at the level of transparency, it is not an additive to recommend.

The results of the XRD analysis show that the samples that present the greatest transparency (15%, kaolin and 5% graphite) are in turn those that show the least quartz crystals in the structure, and the 50/50 mixture is the one that generates the least transparency, and is in turn the one that shows a greater amount of quartz in the structure.

The veracity of what is indicated in the literature was verified that the pre-drying of the moulds reduces the adhesion of the mold material on the surface of the glass as well as improves its transparency, so it is recommended to pre-dry the moulds at 94 ºC, without glass in it, until moisture is no longer perceived in the kiln environment. Depending on the size of the mold, I would dry it between 5 and 10 hours.

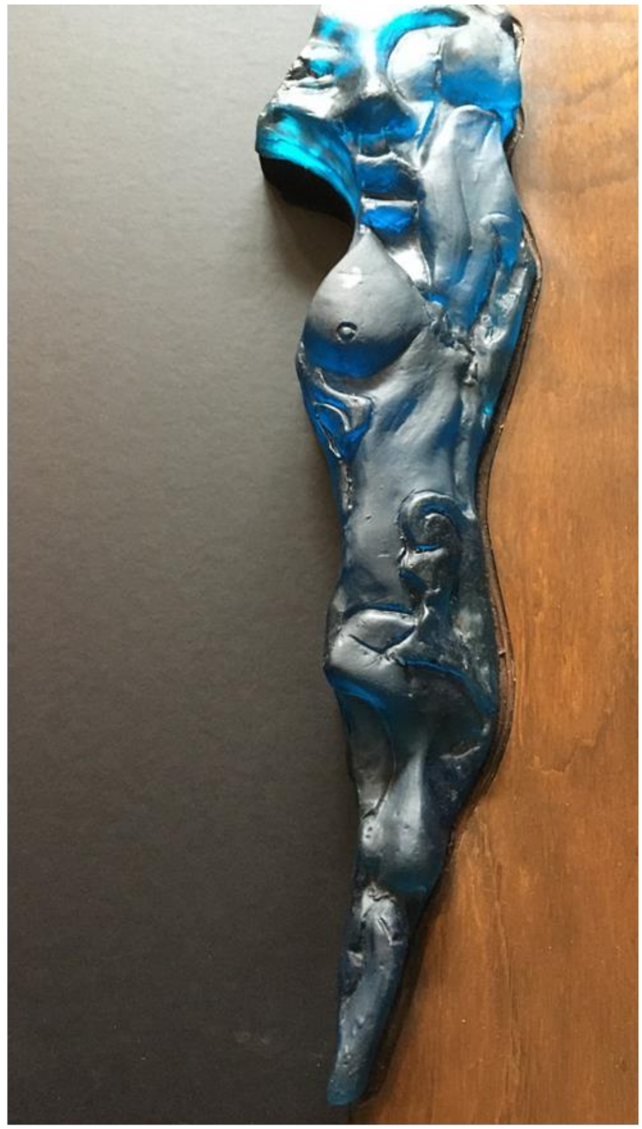
Finally a special effect on the transparency of the glass is not visualized by the use of Shellac, although it works very well to stabilize the mixture avoiding that Plaster particles are trapped in the glass.

Annexes.

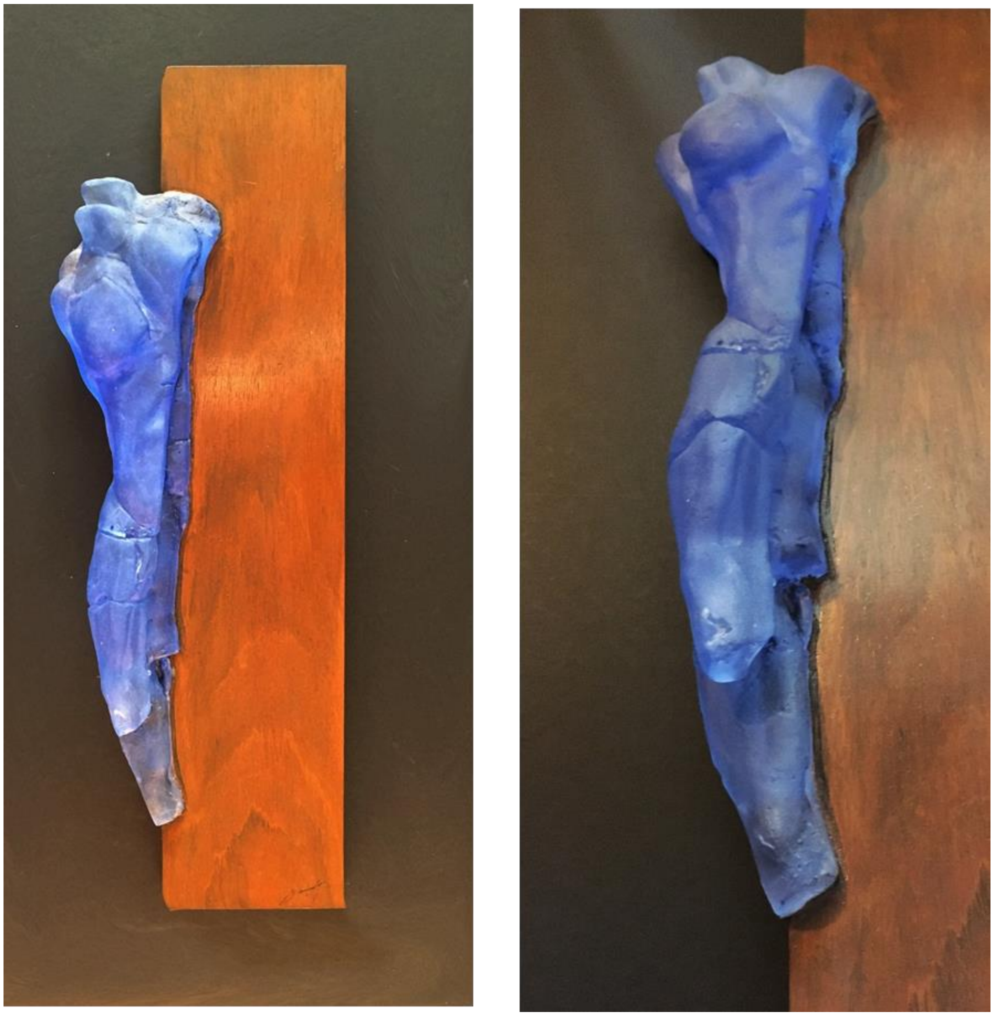
Annex 1: Visual compendium of the art works made for the exhibition

Serie. Encuentros

The pieces show bodies in multiple layers that intertwine and combine to shape the composition. It is a series made in Casting, where the colors are inspired by Portuguese tiles. Wooden bases with simple lines reminding me of the aesthetics of the 70s, the year I was born and which was used in my parents' house in the early years of my childhood.



Encontros 1. Kiln Casting glass, San blasted and hand polished. 2020. 58 x 20 x 4 cm



Encontros 2. Kiln Casting glass, San blasted and oil painted.

2020. 58 x 20 x 4 cm

Encontros 3. Kiln Casting glass, fired polished .
2020. 40 x 20 x 4 cm

Encontros 4. Kiln Casting glass with Kaoiln, oil painted .
2020. 50 x 20 x 6 cm

Encontros 5. Kiln Casting glass with Kaolin, oil painted .
2020. 50 x 20 x 6 cm

Encontros 5. Kiln Casting glass with graphite. 2020. 40 x 7 x 5 cm

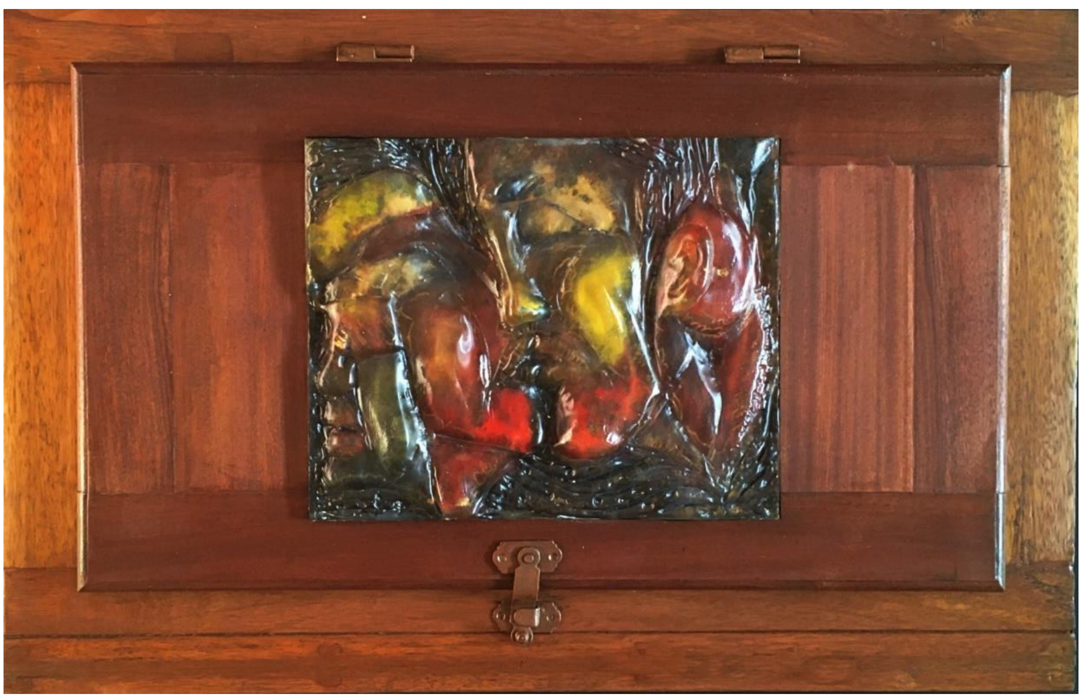
Mi Luna. Kiln Casting glass with graphite, oil painted .
2020. 30 x 30 x 4 cm

Serie. Recuerdos

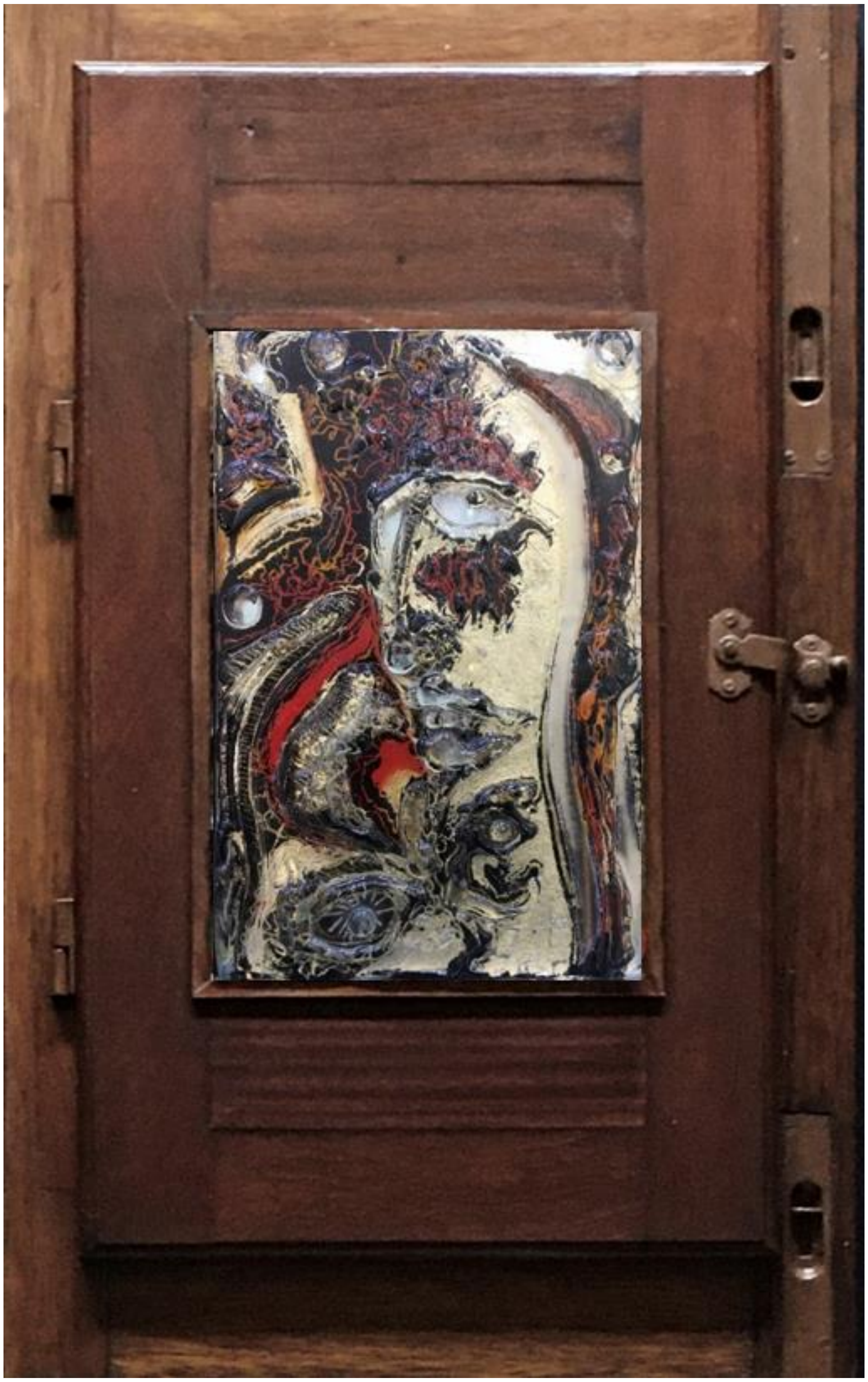
Serie made using recycled window glass, found on the street, giving it a new life, transforming what was discarded by others into art. The use of gold sheets and oil paint with the reverse painting technique, transforms what was for others garbage into an object that exudes luxury and sobriety. It is inspired by the Byzantine mosaics that I met during my stay in Turkey, but with a theme that moves away from the religious vision, showing naked bodies with a sensual and erotic theme. As a frame, the pieces are mounted on restored old windows, contrasting the brightness of the glass with the heat and opacity of the wood.

MyEve. Gilted Fused float glass, with reverse oild painted, on a restaured Wood window.
2020. 29 x 46 x 3.5 cm

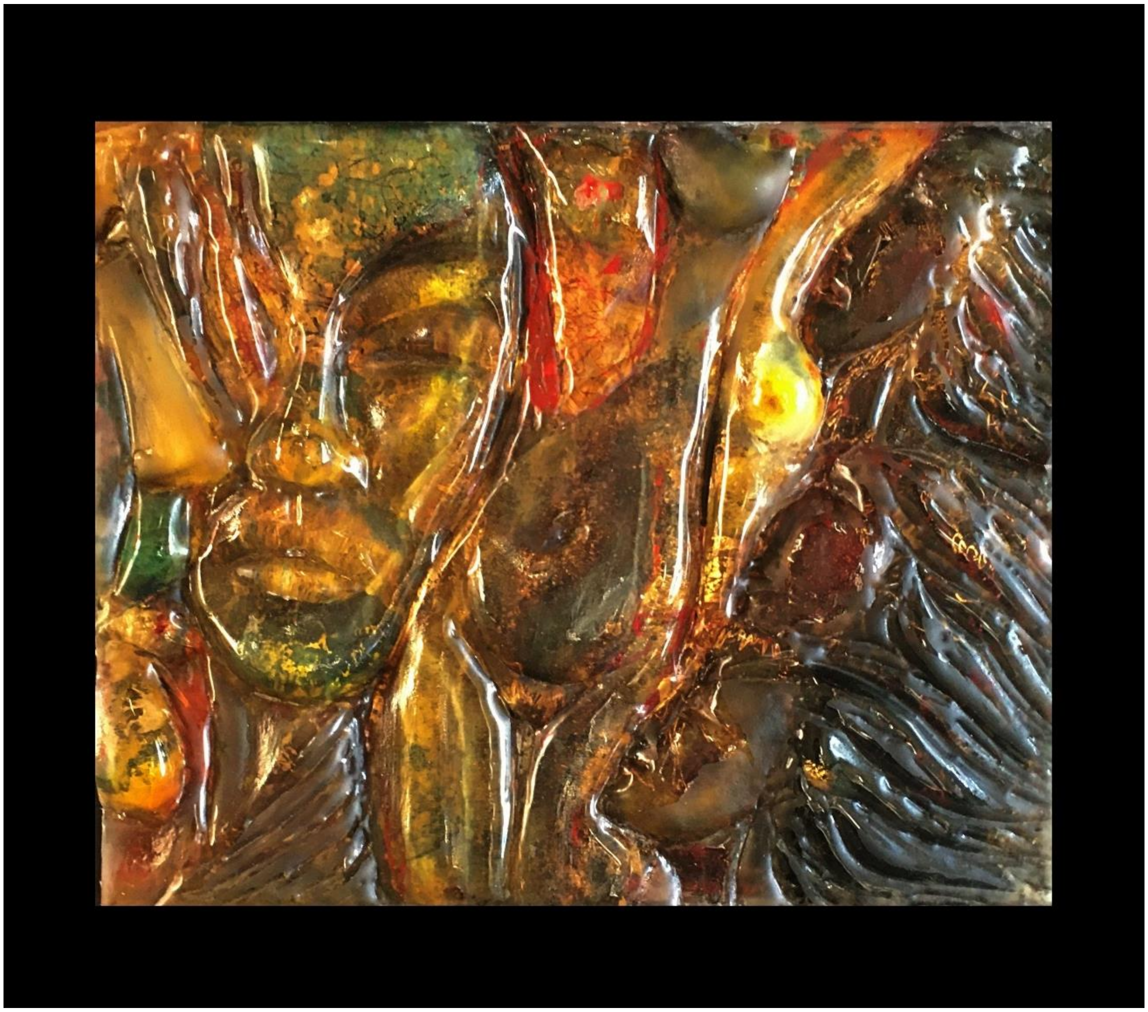
MyAdam. Gilted Fused float glass, with reverse oild painted, on a restaured Wood window. 2020. 29 x 46 x 3.5 cm



The kiss. Gilted Fused float glass, with reverse oild painted, on a restaured Wood window. 2020. 48 x 75 x 3.5 cm



Mirada. Gilted Fused float glass, with reverse oild painted, on a restaured Wood window.
2020. 75 x 48 x 3.5 cm



The Secret. Gilted Fused float glass, with reverse oild painted. 2020. 26 x 32 x 1 cm

Annex 2: Visual Classification of Textured Samples by Glass Type

TABLE 26 VISUAL CLASSIFICATION OF TEXTURED TURKISH GLASS

GLASS	EXAMPLES	BEST	WORST		
		1st	2nd	3th	4th
Turkish Glass With Texture	Ludo: 1 vs 2-4.	4	1	2	3
	Kaolin: 1 vs 5-7,	6	7	1	5
	Talc: 1 vs 8-10.	1	9	8	10
	Graphite: 1 vs 11-13.	11	1	12	13
	Alumina: 1 vs 14-16	1	16	15	14

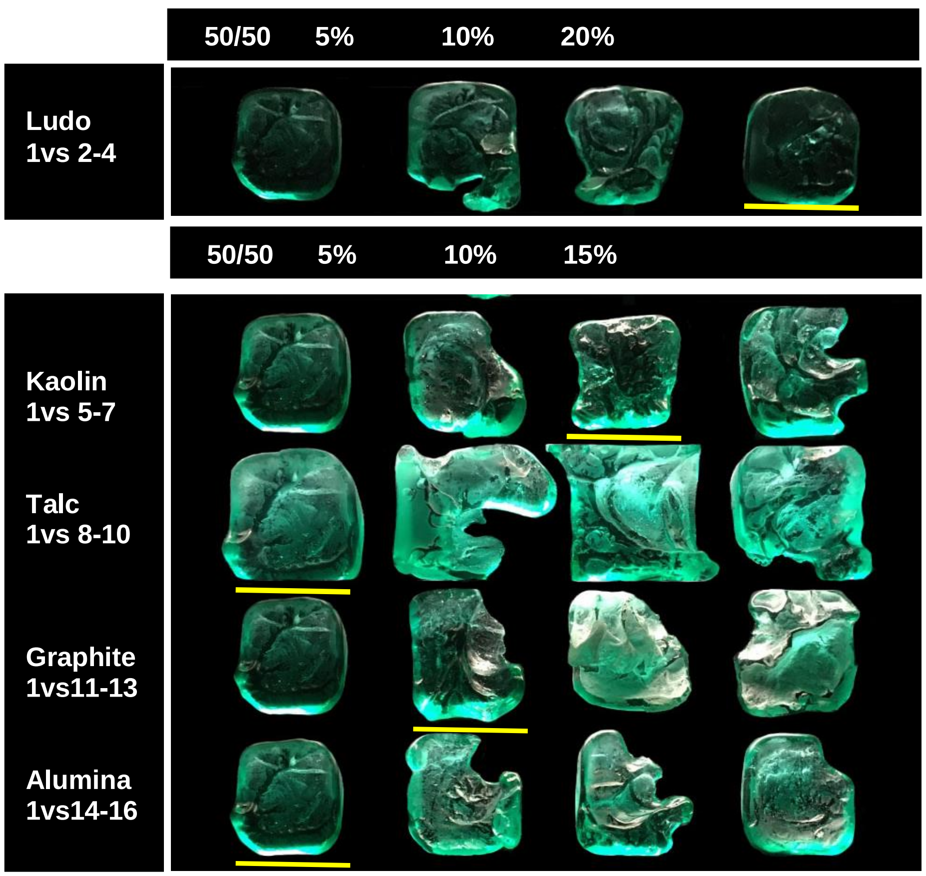


Figure 40. Comparison of textured Turkish glass samples. Those marked with the yellow line represent the best option within each category.

TABLE 27 VISUAL CLASSIFICATION OF TEXTURED FURNACE GLASS

GLASS	EXAMPLES	BEST 1ST	2ND	3TH	WORST 4TH
FURNACE GLASS WITH TEXTURE	Ludo: 1 vs 2-4.	4	3	1	2
	Kaolin: 1 vs 5-7,	7	5	6	1
	Talc: 1 vs 8-10.	10	8	9	1
	Graphite: 1 vs 11-13.	11	12	13	1
	Alumina: 1 vs 14-16	1	15	14	16

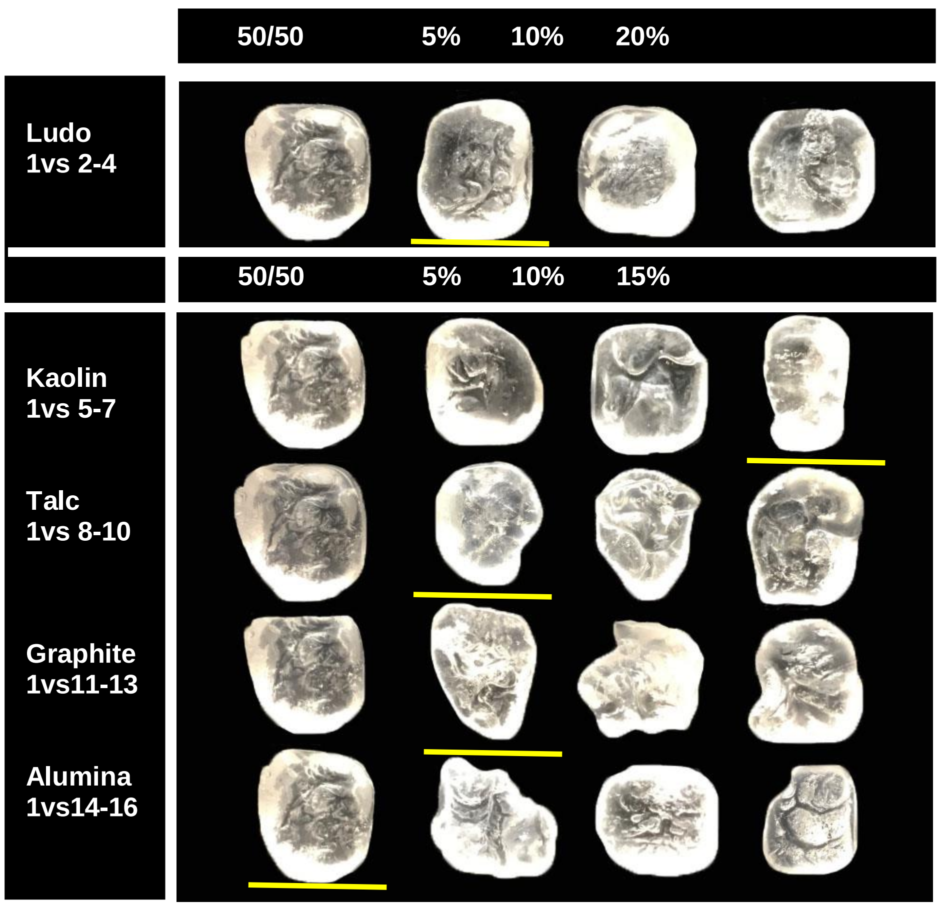


Figure 41 Comparison of textured Furnace glass samples. Those marked with the yellow line represent the best option within each category

TABLE 28 VISUAL CLASSIFICATION OF TEXTURED WINDOW GLASS

GLASS	EXAMPLES	BEST 1ST	2ND	3TH	WORST 4TH
WINDOW GLASS WITH TEXTURE	Ludo: 1 vs 2-4.	1	2	4	3
	Kaolin: 1 vs 5-7,	7	1	5	6
	Talc: 1 vs 8-10.	8	1	9	10
	Graphite: 1 vs 11-13.	11	1	12	13
	Alumina: 1 vs 14-16	1	16	14	15

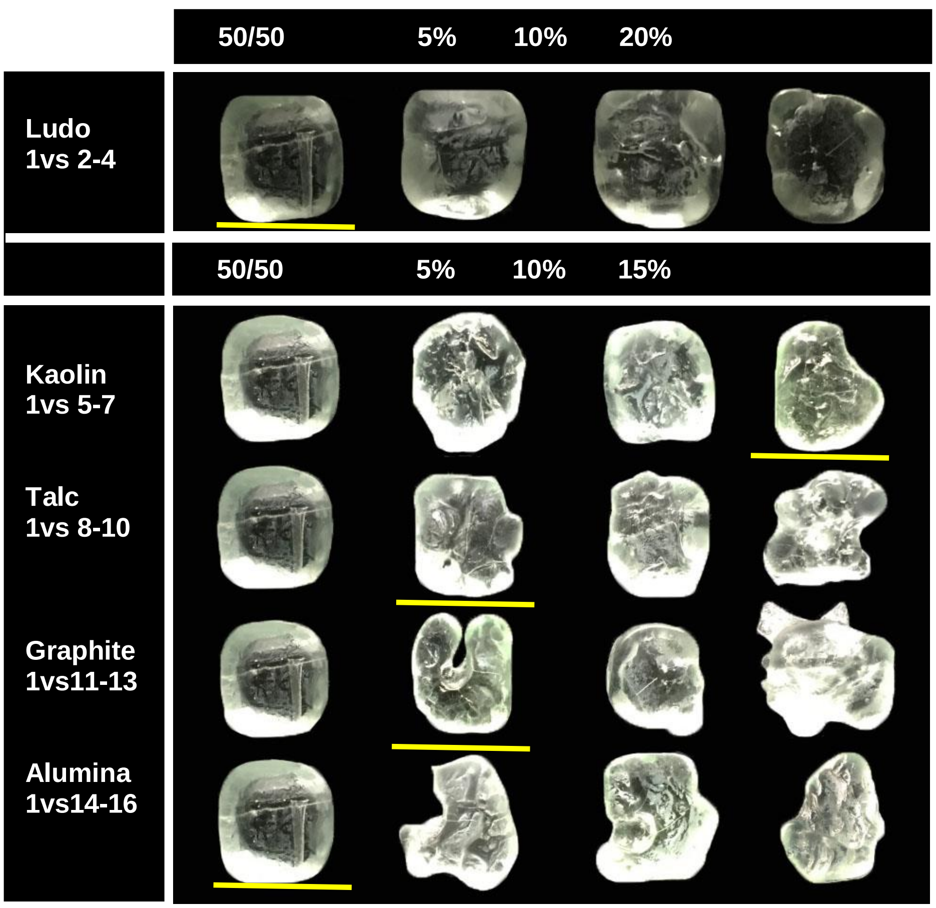


Figure 42 Comparison of textured Float glass samples. Those marked with the yellow line represent the best option within each category

Annex 3: Grouping the samples by Additive

In order to see the effect of each additive in the samples, the results were grouped by additive and ordered, by visual comparison, from best to worst, considering the sample with less residues or devitrification on its surface better. The objective is to see which of the formulas is positioned in the first position. The result indicates that the best formulas are 1 (50/50), 4 (20% Ludo), 7 (15% Kaolin), and 11 (5% Graphite)

TABLE 29 RANKING OF THE BEST FORMULAS BY ADDITIVE

		BEST			WORST
GLASS	**EXAMPLES**	**1ST**	**2ND**	**3TH**	**4TH**
Turkish glass with texture	**LUDO 1VS 2-4**	4	1	2	3
Turkish glass without texture		1	3	4	2
Furnace glass with texture		4	3	1	2
Furnace glass without texture		4	3	1	2
Window glass with texture		1	2	4	3
Window glass without texture		1	4	3	2

		BEST			WORST
GLASS	**EXAMPLES**	**1ST**	**2ND**	**3TH**	**4TH**
Turkish glass with texture	**KAOLIN 1VS 5-7**	6	7	1	5
Turkish glass without texture		5	6	7	1
Furnace glass with texture		7	5	6	1
Furnace glass without texture		7	5	6	1
window glass with texture		7	1	5	6
window glass without texture		7	5	6	1

		BEST			WORST
GLASS	**EXAMPLES**	**1ST**	**2ND**	**3TH**	**4TH**
Turkish glass with texture	**TALC 1VS 8-10**	1	9	8	10
Turkish glass without texture		8	1	9	10
Furnace glass with texture		10	8	9	1
Furnace glass without texture		9	1	8	10
window glass with texture		8	1	9	10
window glass without texture		1	8	10	9

		BEST			WORST
GLASS	**EXAMPLES**	**1ST**	**2ND**	**3TH**	**4TH**
Turkish glass with texture	**GRAPHITE 1 VS 11-13.**	11	1	12	13
Turkish glass without texture		11	1	12	13
Furnace glass with texture		11	12	13	1
Furnace glass without texture		11	12	1	13
window glass with texture		11	1	12	13
window glass without texture		11	1	12	13

		BEST			WORST
GLASS	**EXAMPLES**	**1ST**	**2ND**	**3TH**	**4TH**
Turkish glass with texture	**ALUMINA 1 VS 14-16**	1	16	15	14
Turkish glass without texture		15	14	1	16
Furnace glass with texture		1	15	14	16
Furnace glass without texture		1	14	15	16
window glass with texture		1	16	14	15
window glass without texture		1	14	15	16

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.Annex 4: Calculation of the best 4 formulas

In order to obtain the 4 best formulas to continue the experimentation, its frequency in each quality position is calculated, scoring from 4 to 1, from highest to worst, and the total is calculated thus giving the relative position of each formula concerning the quality of the result. The first position receives 4 points, the second 3 and so on.

For example, experiment 1 was in the first position in three of the samples, one in the second position, 2 in the third and once in the fourth. The calculation would be as follows: 3 * 4 + 1 * 3 + 2 * 2 + 1 * 0: 19, giving as a result that for the use of Ludo the formulas that generate the greatest transparency are 1 and 4. The result indicates that the best 4 formulas are 1, 4, 7 and 11

TABLES 30 RANKING OF FORMULAS BY FREQUENCY IN THE POSITIONS FROM BEST TO WORST

Samples Ludo		**BEST**	**WORST**			
		1st	**2nd**	**3th**	**4th**	**total**
	Points	**4**	**3**	**2**	**1**	
1	Frequency	3	1	2	0	19
2		0	0	3	3	9
3		0	2	1	3	11
4		3	1	2	0	19
		4	**1**	**3**	**2**	

Samples Kaolin		**BEST**	**WORST**			
		1st	**2nd**	**3th**	**4th**	**total**
	Points	**4**	**3**	**2**	**1**	
1	Frequency	0	1	1	4	9
5		1	3	1	1	16
6		1	1	3	1	14
7		4	1	1	0	21
		7	**5**	**6**	**1**	

Samples Talc		**BEST**	**WORST**			
		1st	**2nd**	**3th**	**4th**	**total**
	Points	**4**	**3**	**2**	**1**	
1	Frequency	2	3	0	1	18
8		2	2	2	0	18
9		1	1	3	1	14
10		1	0	1	4	10
		1	**8**	**9**	**10**	

Samples Graphite		**BEST** **WORST**				
		1st	**2nd**	**3th**	**4th**	**total**
	Points	**4**	**3**	**2**	**1**	
1	Frequency	0	4	1	1	15
11		6	0	0	0	24
12		0	2	4	0	14
13		0	0	1	5	7
		11	**1**	**12**	**13**	

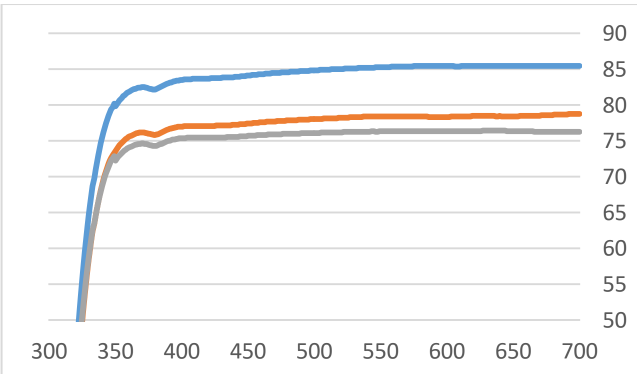
Samples Alumina		**BEST** **WORST**				
		1st	**2nd**	**3th**	**4th**	**total**
	Points	**4**	**3**	**2**	**1**	
1	Frequency	4	1	1	0	21
14		0	3	2	1	14
15		1	1	3	1	14
16		1	1	0	4	11
		1	**14**	**15**	**16**	

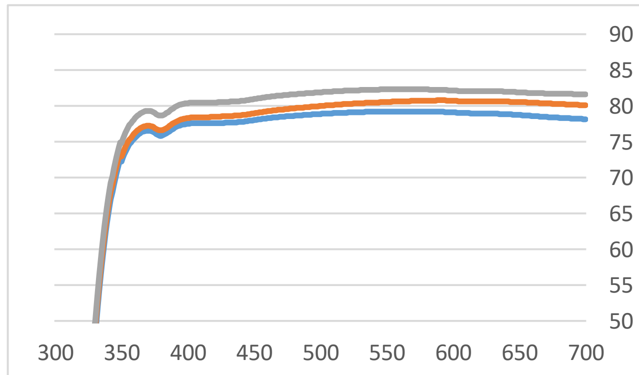
Annex 5: Comparison between the Transmittance of Ludo and Graphite

Transmittance was measured at three different locations in the samples. As can be seen in the graphs, Ludo has a greater range of variation than graphite, which translates into less homogeneity on the glass surface.

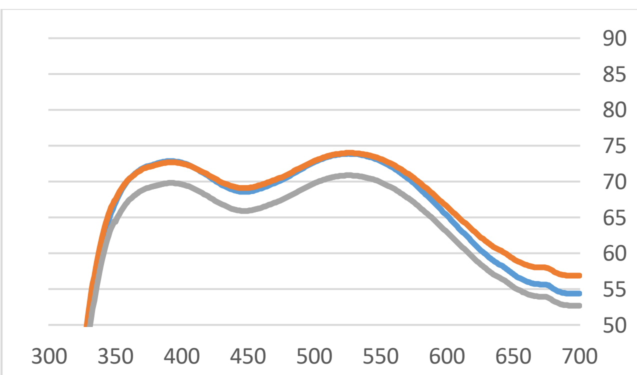
LUDO GRAPHITE

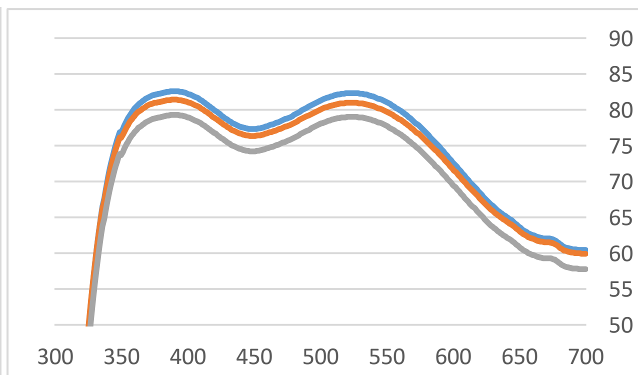
FLOAT



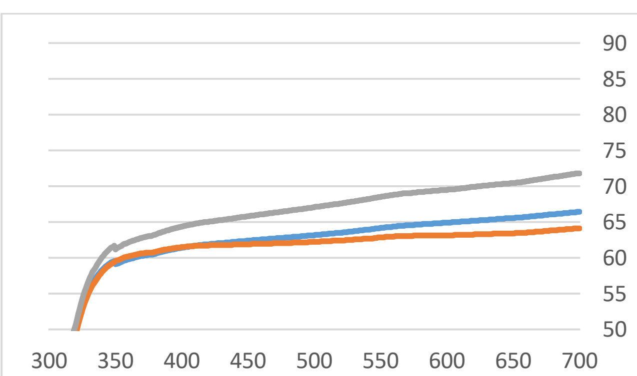


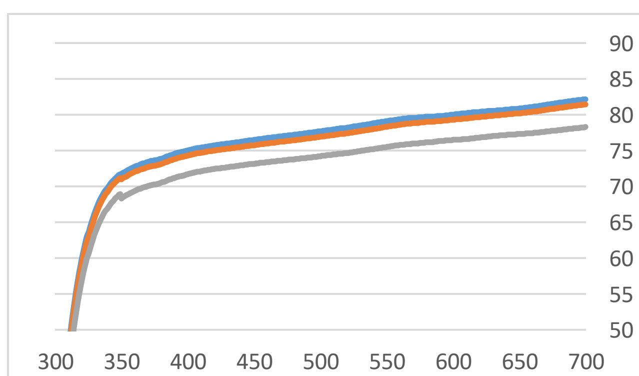
TURKISH





FURNACE





Annex 6: Some of Investment for casting available on the market

TABLA 31 PREMIX INVESTMENT AVAILABLE IN THE MARKET

NOMBRE	DESCRIPTION
KS-4	Dense, Strong, and General-Purpose Castable Refractory for Temperatures up to 2550 ° F Features: • Good strength.
Glass-Cast™ 910	Strongest mold material available for glass casting on the market today and is considered the glass caster's best choice for large pieces
Glass-Cast™ 400	Stronger, economical mold material. It provides added strength for the glass artist that struggles with mold integrity due to mold size or process intensity
Glass-Cast™ 101	Glass-Cast 101 BANDUST investment is an economical, all-purpose mold material for kiln casting/slumping of glass. With very fine particles
Mould Mix 6	Refractory-molding compound designed to allow replication of three-dimensional objects in glass. Properly prepared moulds of Mold Mix 6 will resolve the finest details and possess good strength. However, it remains sufficiently friable to permit easy removal after annealing. Mold Mix 6 is highly resistant to reaction with hot glass and gives the fired work a high quality surface free of-hazing common to investment type moulds
A.P. Green KOL26LI - Kast-O-Lite	

Annex 7: Turkish Glass Composition

TABLA 32 TURKISH GLASS TECHNICAL DATA. ÇALIŞKAN KRISTAL COMPANY

Abbe's number	vd=58.852, ve=58.627
Hydrolytic class	HGB 3
Alkaline class according to ČSN ISO 695	A2
Acidic class according to DIN 12116	1.

standard length of rod	1120 ± 10 mm
the standard package weight (one bundle)	20 kg
transport package	
bundles in cases on wooden pallet	600 kg ntto, 650 kg btto
free loaded in one case on wooden pallet	700 kg ntto, 750 kg btto

Informative composition in %			
SiO_2	68	CaO	6
Na_2O	11	BaO	4
K_2O	6	ZnO	3

Physical and chemical characteristics		
specific gravity	ρ	2,585 kg/m^3
coefficient of expansion	α	$9,4 \pm 0,4 . 10^{-6} K^{-1}$
index of refraction	n_D	≥1,520
softening point ($10^{7,65}$ dPa s)	t_L	686 ± 10 °C
transformation point ($10^{13,3}$ dPa s)	t_g	504 ± 10 °C
deformation point(10^{11} dPa s)	t_d	568 ± 10 °C

group 5
Emerald 50xx

$SiO2$ 72%, $Na2O$ 12%, $K2O$ 9%, CaO 4%, BaO 3%
T_d 645 °C T_l 540 °C T_g 473 °C α =10,21 x 10

group 6
Jantar 800x, Ruby 900xx

$SiO2$ 67%, $Na2O$ 12%, $K2O$ 9%, CaO 1%, MgO 1%, BaO 1%, ZnO 5%, $Al2O3$ 1%, $B2O3$ 3%
(coloring: CdO + Se) T_d 654 °C T_l 524 °C T_g 460 °C α =10,12 x 10

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