# High Pressure Reactor Material Selection for Microwave-assisted Leaching of Platinum Group Metals

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**Abstract- The Colombian Platinum floods are made up of platinum, iridium, osmium and palladium. Iridium and Osmium confer a refractoriness condition that makes alluvium leaching difficult, being the first aspect to be solved in the development of a refining process. Microwave acid leaching offers a higher leaching rate with high levels of recovery, so it seems a possible process to move forward in this regard. For this, a reactor with microwave reception capacity has been designed, with a volume of 180 ml at low cost and that meets the requirements of pressure, temperature, chemical resistance and safety. In the first phase the possible acid mixtures were determined by means of the literature, looking for the most effective one with pressure and temperature characteristics. A study was then carried out on the different reactor designs that exist at the international level of companies (Parr, CEM corp) and an easy-to-build one was proposed in our environment. The mechanical calculations of the reactor were performed according to ASME code section VIII. Subsequently, the material is selected using the Ashby methodology and Ces-EduPack software, which resulted in PTFE and POM-H.**

**Keywords – reactor; leaching; temperature; pressure.**

## 1. INTRODUCTION

Platinum (Crundwell et al., 2011) is considered a noble metal, that is, resistant to chemical attack; However, it is one of the most reactive heavy metals, it is used for medicine (Oscar Aguilar Cuevas, Rodrigo Castro Ramírez, Jessica J. Sánchez García, Horacio López-Sandoval, 2012), as a catalyst in the automobile industry (Jafarifar et al., 2005), chemical industry, electricity and electronics, glass manufacturing, petroleum refining and biomedical. In Colombia, the refining of the platinum group is not carried out since the necessary technology is not available, therefore, mining companies take this metal raw, because the processes are complicated and involve complex elements such as dissolution, channeling and precipitation, hydrolysis, distillation, organic precipitation, solvent extraction, ion exchange, molecular recognition technology and metal reduction, among others (Edwards 1976). The recovery of platinum, palladium, rhodium and ruthenium (Suoranta et al., 2015) by means of microwave assisted leaching is a faster and therefore more energy efficient way to dissolve these elements. It is also possible to obtain, in most cases; a recovery of 100% in a single stage, which saves time in the process (Alguacil, 1995).

Of selection of a material that will be the perfect fit for the design of a component that complies with the requirements or specifications determined during the design phase. The settings depend a lot on the design, and can be: cost, weight, chemical properties and/or behavior in the environment (humidity, contact with salt water, acidic environment, etc.), mechanical properties (tensile strength, compression stress, flexural modulus etc.), ecological properties (recyclable, reusable, biodegradable, carbon footprint) etc. Although there are many methods for design setting optimization and for the selection of materials, the Ashby methodology is one of the few that integrates mechanical factors, physical and chemical properties with ecological ones, and in turn, manufacturing and amalgamation technologies (Ashby & Cebon, 2005; M. F. Ashby, 2013).

Currently the number of available materials (metals, polymers, ceramics, composites, foams etc.) is very wide, more than 5000. For this, the designer has the time-consuming task of selecting the most appropriate material for his design from this wide range of possibilities. This selection can be done most easily by using the method proposed by Michael Ashby, which relates the design specifications in a catalogue of materials. Additionally, Ashby defines a material index as a grouping of mechanical properties that, if maximized or minimized, enhance some aspect of the performance of an engineered component. The Ashby method achieves this objective through 4 steps that are outlined in Figure 1.

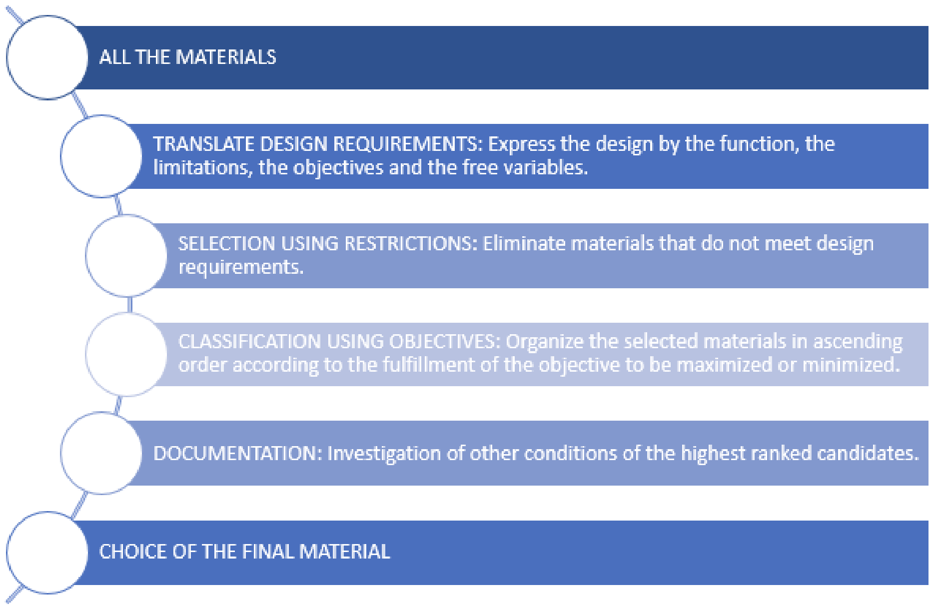


Figure 1. Ashby methodology for the selection of materials.

*1.1) Translating design requirements.*

At this stage, the design specifications must be "translated" from the normal language that is commonly used, to the technical language in terms of specific properties with quantitative or qualitative units. For this stage, Ashby proposes: for it to be developed in 4 steps (function, restrictions, objectives and free variables), in which the following specific questions are answered:

**Function**
What does the component do?
**Restrictions**
What requirements must be met and are they NOT negotiable in the design?
**Objective**
What requirements should be maximized or minimized?
**Free variable**
What design parameters are they free to choose and do not affect the above?

The function is understood as the purpose of the component; this answers the question in the chart above. The product of the translation is a specific property of the material. Design constraints or limitations, which can be static dimensions, specific stresses, specific mechanical settings, chemical or physical properties, etc. These are properties that are not a function of any part of the component that is being designed, they are necessary for the design to be viable, however; these are properties that cannot be negotiated. The objectives are settings or specifications whose effectiveness you want to maximize or minimize.

*1.2) Selection using restrictions.*

At this stage, from the entire "universe" of possible materials, or those defined according to the design (Ceramics, polymers, metals, etc.), an impartial selection is made between the candidates who comply with the different aforementioned restrictions. The process selection discards materials that do not meet the requirements established in the restrictions, meaning that their attributes are outside the established limits. This can be done graphically using the CES EDUPACK software. At this stage it is also necessary to determine the index of the material. The material index is a mathematical expression that relates the property that has been established as the main one in the design, this is called a function, and it is related to the property that you want to maximize or minimize.

*1.3) Prioritization using objectives.*

Once the possible materials have been selected, one (1) selection criterion is used, previously mentioned in the first stage as the objective. It is then prioritized in an ascending or descending way, as required in different circumstances.

*1.4) Documentation.*

The selected materials are put through an investigation process to determine the availability of the material in the region, distribution channels, transportation and delivery times, experience and support of the manufacturers, etc. This type of additional information can often tip the balance towards the choice of a particular material.

*1.5) Choice of final material.*

With this information, the material to be used for the specific purpose is determined.

## 2. EXPERIMENTAL

In the design of the leaching reactor, it was carried out as follows.

*2.1) Design.*

For an economical and easy-to-build design the following model was carried out, it can be seen in figure 2. This design has:

- Reactor, where the leaching process occurs with a capacity of 180 ml.
- Reactor top.
- Design of a seal of confinement with static conditions, which will allow a hermetic closure of the container and the top.
- Sleeve, designed to provide the mechanical properties necessary to withstand the design pressure. A fine pitch metric thread is designed on the sleeve for closure.
- Jacket top.
- This compression disc, alongside the cap threaded to the sleeve, will allow the closure between the reactor and its cap.

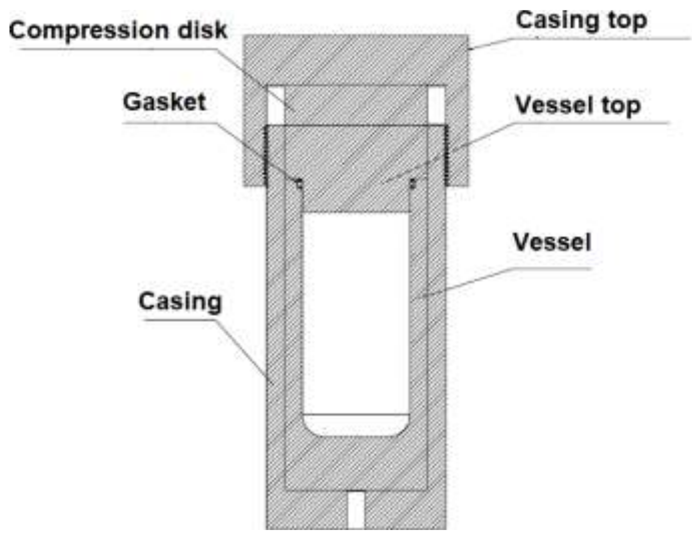


Figure 2. Reactor design.

3. RESULTS

*3.1) Design parameters*

The materials are established to be transparent to microwaves, these exhibit excellent resistance to aqua regia, and a resistance to high temperatures greater than 250 ° C (Parr, 2005; Plus, n.d.). Also, it possesses high mechanical properties that allow it to withstand up to 1200 psi (Parr, 2005). Chart 1 shows the properties necessary to select the material according to the Ashby method. In addition, a material that is easy to machine is required. The material index is established according to the requirement of it being a cylinder with internal pressure, seeking to maximize the yield stress (ASHBY & CEBON, 1993).

Chart 2 shows the necessary properties for the materials needed for the liner and top.

Chart 1. Ashby methodology for reactor and top.

| | **Common** | **Property** | **Sign** | **Value (α-#)** |
|---|---|---|---|---|
| **Function** | Resistant to high pressures. | Yield strength | ≥ | 10 MPa* |
| **Restrictions** | Resistant to high temperatures. | Max temp. of service | ≥ | 250 ° C |
| | Resistant to strongly oxidizing environment | Royal water resistance | ≥ | Excellent. |
| | Transparent to microwave | Transmissivity | ≥ | Polymers, ceramics and composites. |
| | Make it machinable | Machinability | ≥ | Metals, polymers and compounds |
| | Do not burn | Inflammability | ≤ | Not flammable |
| **Objective** | Economic<br>Lightweight<br>Do not make an arc with the microwave. | Price<br>Density<br>Electric conductivity | | Minimize<br>Minimize<br>Minimize |
| **Material index** | $\sigma_f/\rho$ | | | |

* A reverse engineering process was performed to determine this value.

Chart 2. Ashby methodology for the shirt and the cap of the same.

| | Common | Property | Sign | Value (α-#) |
|---|---|---|---|---|
| **Function** | Resistant to high pressures. | Yield strength | ≥ | 50 MPa |
| **Restrictions** | Transparent to microwave<br><br>Make it machinable<br><br>High temperature resistance | Transmissivity<br><br>Machinability<br><br>Maximum service temperature<br><br>Inflammability | ≥<br><br>≥<br><br>≥<br><br>≤ | Polymers, ceramics and composites<br><br>Metals, polymers and compounds<br><br>90 ° C<br><br>Not flammable |
| **Objective** | Economic<br>Lightweight<br>That does not arch with microwaves. | Price<br>Density<br>Electric conductivity. | | Minimize<br>Minimize<br>Minimize |
| **Material index** | $\sigma_f/\rho$ | | | |

*3.2) Selection of materials:*

Once the properties are defined as specified in the previous charts, the CES-EduPack software is used, here the mechanical, thermal, and chemical properties necessary for the reactor are designed (Granta Desing, 1999), the screen out process is carried out and the ranking of materials that fulfill with both the functions and the restrictions is performed. Finally, the material index is used to define which would be the ideal material. It is important to take into account the availability of these materials in the local market or environment.

- Reactor and reactor top.

Four graphs are made; these are shown below. Figure 3 shows the result of the maximum service temperature and the yield point a selection of values ranging between 250 to 330 ° C and 10 to 50 MPa respectively must be made; in figures 4 and 5 the result of resistance to aqua regia, nitric acid, hydrochloric acid, and flammability the materials must be reduced to 4 candidates; finally, in figure 6 the density and the price range can be appreciated, as well as the search process keeping the lowest values of the aforementioned in mind.

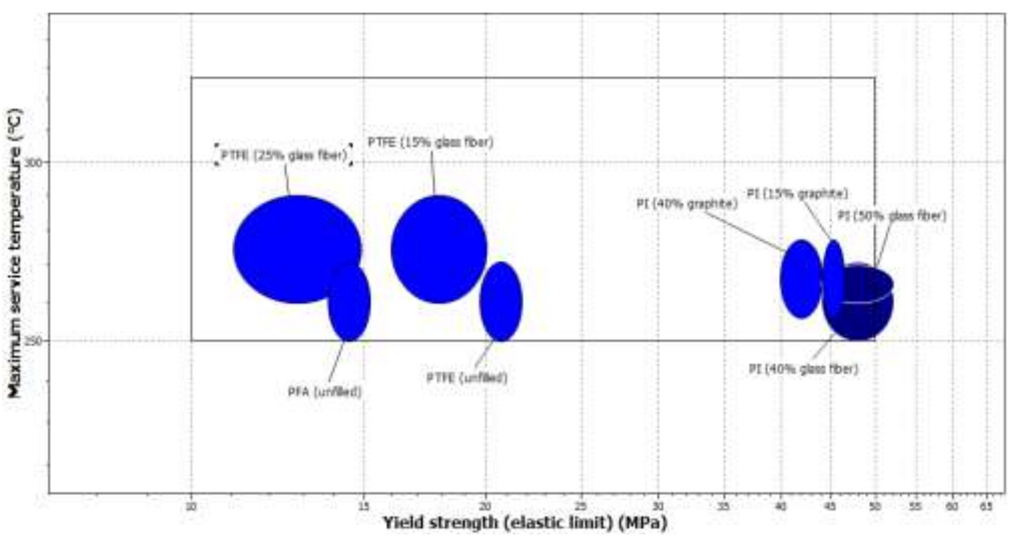


Figure 3. Reactor body and top - Maximum service temperature Vs. yield strength.

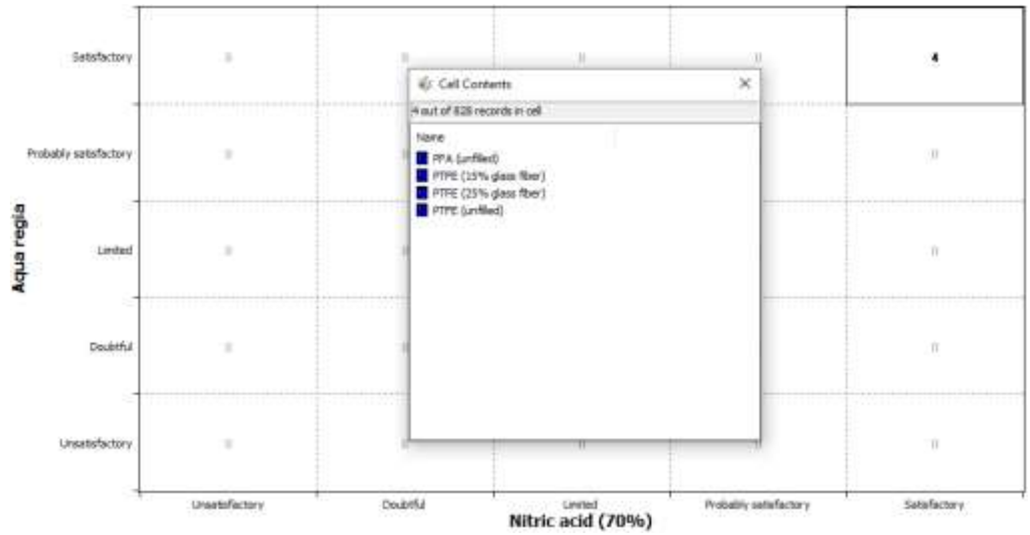

Figure 4. Reactor body and top. Aqua regia Vs. Nitric Acid 70%.

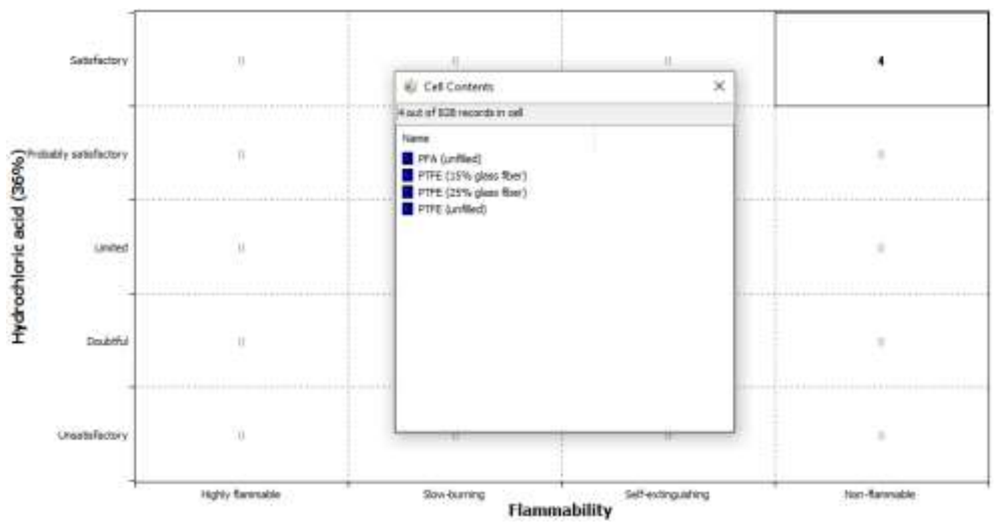

Figure 5. Reactor body and top. Hydrochloric acid Vs. Flammability.

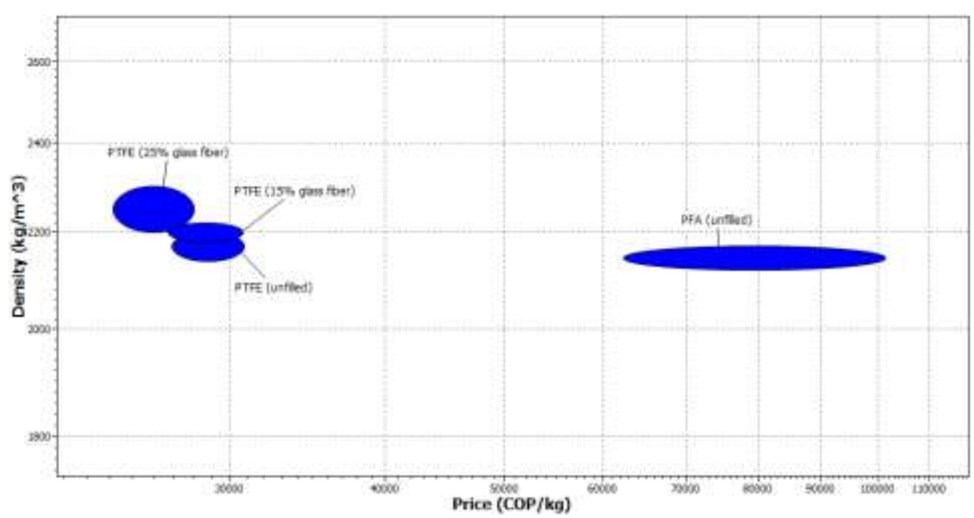

Figure 6. Reactor body and top. Density Vs. Price.

The resulting materials in the intersection of the figures are:

- ✓ Perfluoroalkoxy ethylene (PFA).
- ✓ Polytetrafluoroethylene 15% fiberglass (PTFE 15% fiberglass).
- ✓ Polytetrafluoroethylene 25% fiberglass (PTFE 25% fiberglass).
- ✓ Polytetrafluoroethylene (PTFE).

The material index allows the maximization of the property that is required in the design, being the yield stress and minimization of cost and density, in figure 7 this can be seen aligned with slope of 1, chosen accordingly to such index. This line allows you to find the best ratio of the highest mechanical property and the lowest price and density.

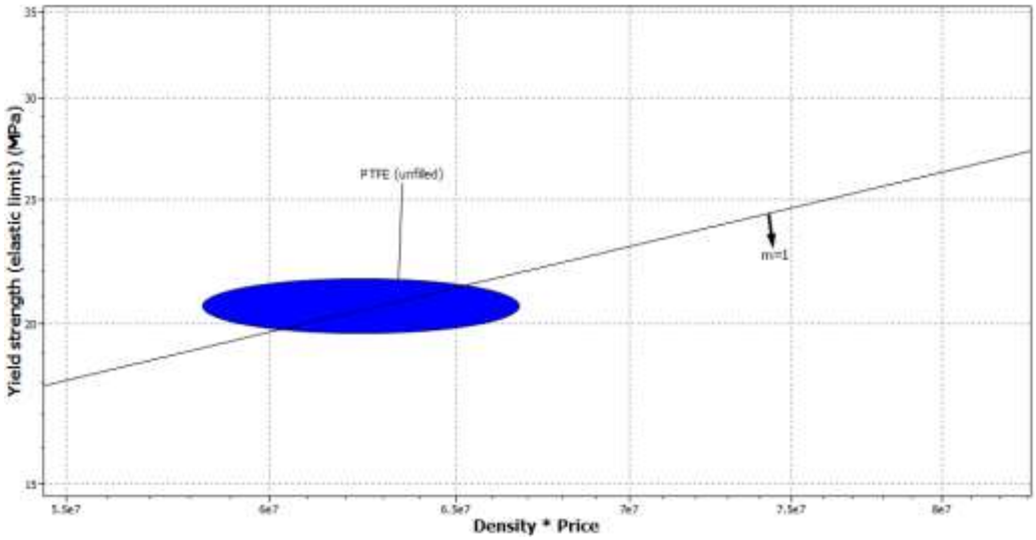


Figure 7. Body and shirt top. Yield stength Vs. Density * Price.

Complying with the mechanical, thermal, and chemical properties and also using the material index defined for this project (chart 1), the PTFE (unfilled) (figure 7) is selected for the construction of the reactor and its top, this material is commonly called Teflon and is considered a thermosetting polymer (COPLASTIC, 2019) (Friction & Energy, 2017) (P.J. & E.N., 2005).

- Jacket and jacket top

For this subsystem, higher mechanical properties are required, in addition, a fine pitch metric thread design to contain the maximum working pressure while keeping in mind properties such as resistance to tension, creep, and shear. In figure 8, compression resistance Vs. yield strength is plotted selecting values from 50 to 120 MPa and 50 to 100 MPa respectively; figure 9 shows maximum service temperature vs. tensile strength with values of 90 to 200 ° C and 50 to 120 MPa respectively, lastly; figure 10 shows density and price and the values that require minimization.

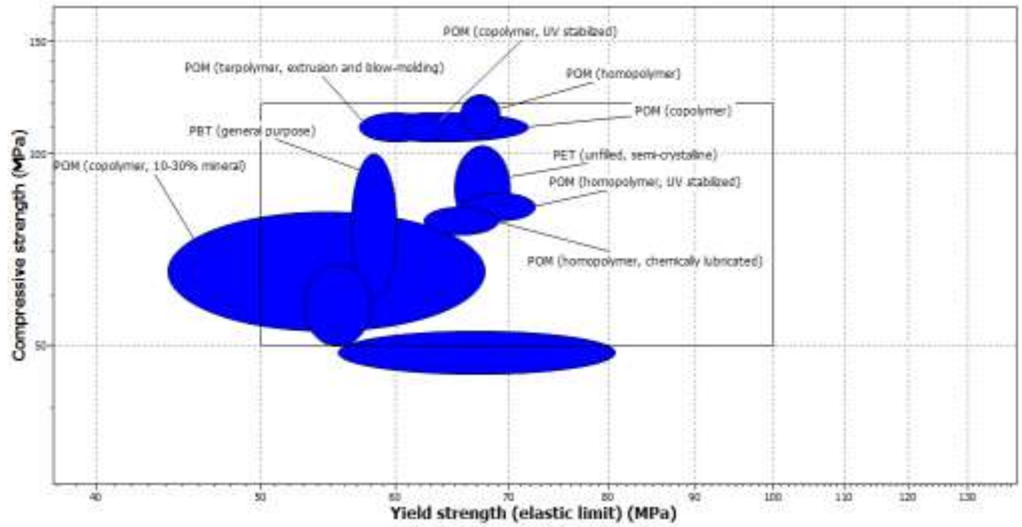


Figure 8. Body and shirt top. Compressive strength Vs. yield strength.

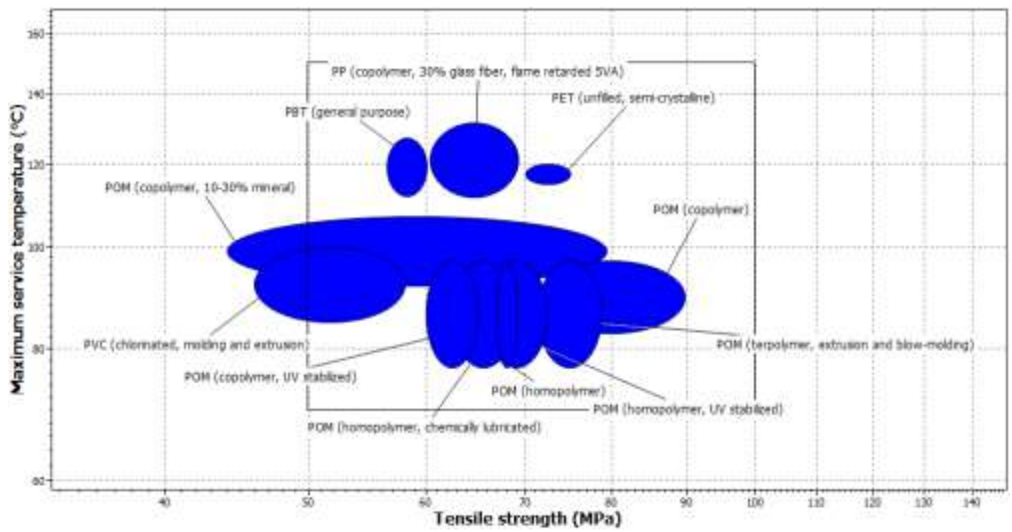


Figure 9. Body and shirt top. Maximum service temperature Vs. Tensile strength.

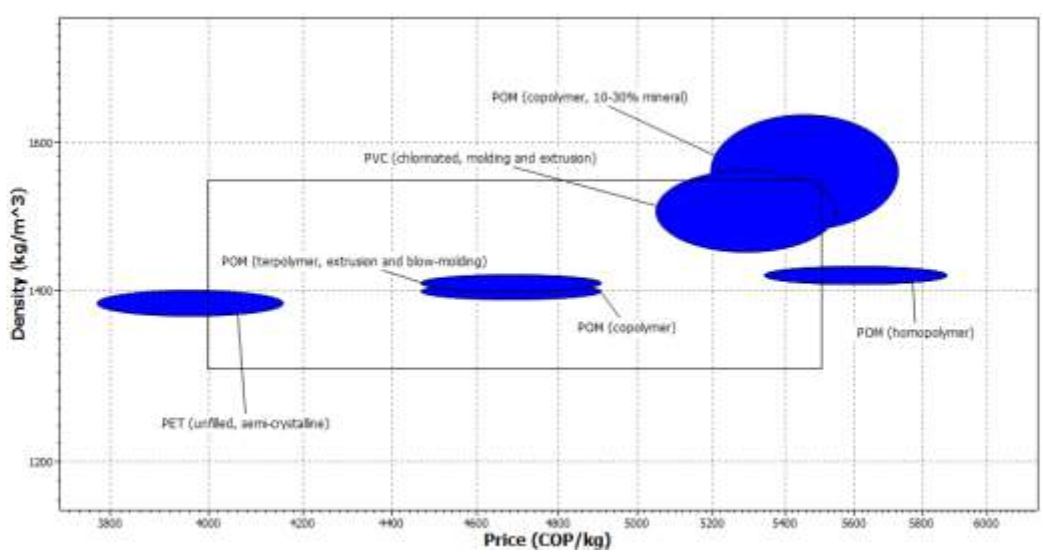


Figure 10. Density Vs. Price.

The resulting materials are:

- ✓ PET (unfilled, semi-crystaline).
- ✓ POM (copolymer)
- ✓ POM (copolymer, UV stabilized)
- ✓ POM (terpolymer, extrusion and blow-molding)
- ✓ PVC (chlorinated, molding and extrusion)
- ✓ POM (homopolymer, UV stabilized) POM (homopolymer)
- ✓ POM (homopolymer, chemically lubricated)
- ✓ POM (copolymer, 10-30% mineral).

The choice of material is determined using the material index, where it is sought to maximize the yield stress and minimize the density and precision relationship. In figure 11 we can see it aligned with a slope of 1, chosen accordingly to this index.

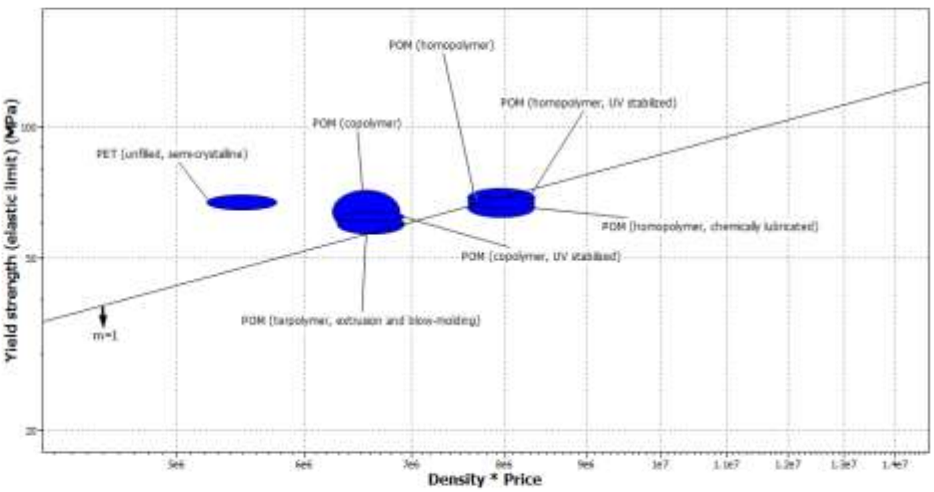

Figure 11. Body and shirt top. Yield strength vs. density * Price.

The material selected for the liner and cap is POM-H homopolymer polyacetal, because it is the easiest one to acquire in Colombia. This is a material that meets the mechanical properties and the purpose for which the design is required (Polytech S.A.S, 2019) (Wyatt Hargett, JR. & ; David Barclay, 2011).

*3.3) Manufacture of the reactor.*

The reactor was manufactured using CNC by numerical control, this allows for greater precision in measurements with tolerances of ± 0.1 mm. The reactor set and the jacket were put together using a loose configuration, in a free operation adjustment H9/d9, this being recommended for high temperatures and pressures. The plug and reactor kit has a localized H7/h6 loose adjustment allowing a tight fit with the advantage of being freely assembled and disassembled (Budynas & Nisbett, 2008). Figures 12 to 14 show the parts of the reactor.

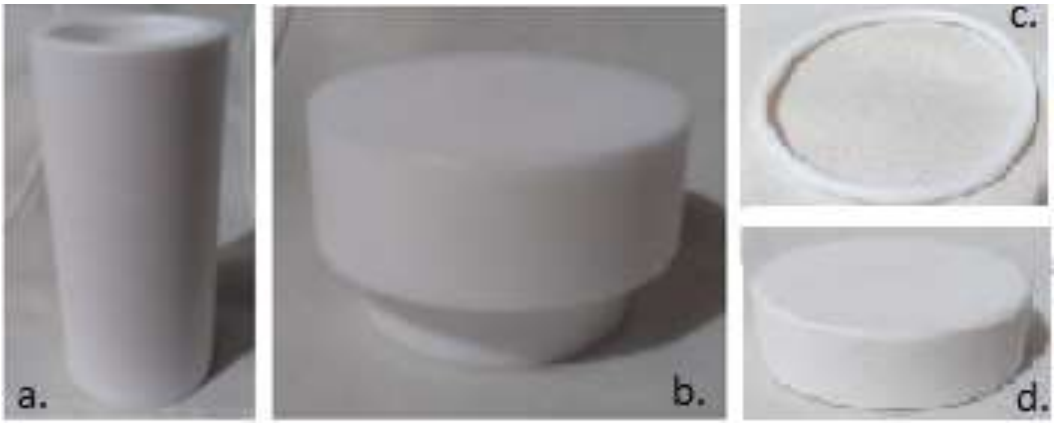

Figure 12. Reactor a.- Body, b.- Top, c.- O-ring, d.- Compresion disk.

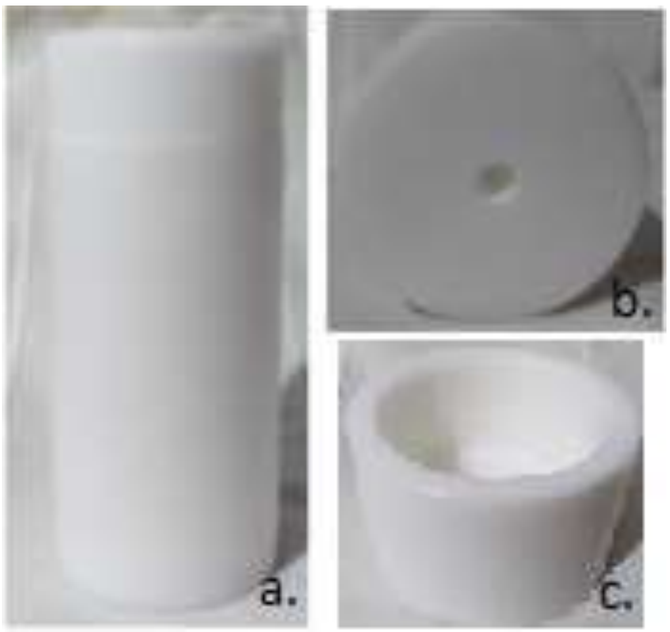

Figure 13. Jacket a.- Body, b.-Compresion disk, c.-Top


Figure 14. Reactor.

*3.4) Reactor test.*

For the reactor test, a Samsung Microwave was used, with an input power of 1000 W. The reactor was charged with 60 ml of dyed water, as seen in figure 15a. Points were marked on the body of the jacket as shown in figure 19b to take temperature measurements using a Fluke brand digital pyrometer. The reactor was sealed, and placed in the microwave (see figure 15c), for 2 minutes.

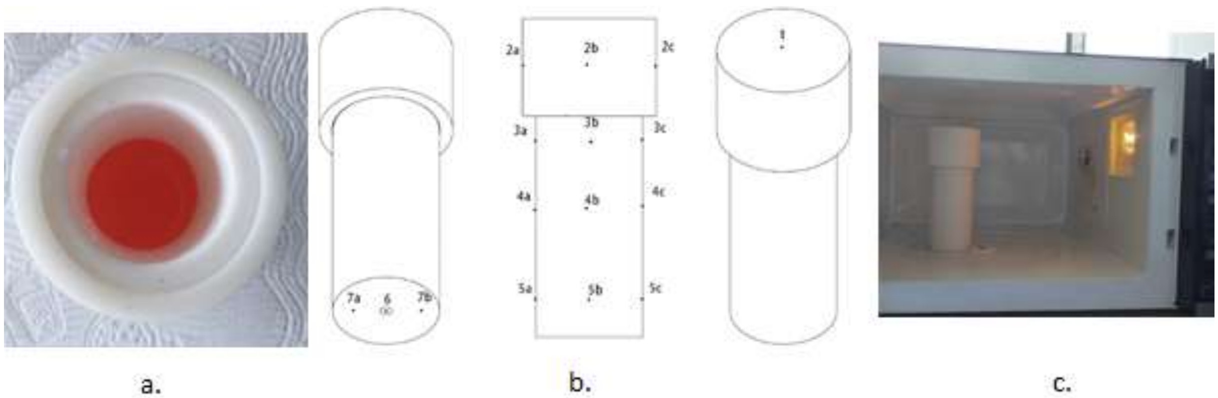

Figure 15. a.- Reactor with 60ml of water; .b.- Temperature points; c.- Reactor inside the microwave.

In figure 16 the temperatures reached in the wall of the jacket can be observed once the test was carried out. As can be seen, the highest temperature is reached in section 5, where the liquid is present inside the reactor and the heating process occurs. On the other hand, it is interesting to note that the radial temperature difference was negligible. The reactor top has a high temperature because a last-minute gasket was added to facilitate the opening of the cap, this gasket is not resistant to high temperatures, it is a nitrile rubber with mechanical and thermal properties lower than those of the PTFE and POM-H. Furthermore, it can be verified that the temperature reached is well below the maximum service temperature of the POM-H, which is 150 ° C for short periods (Polytech S.A.S, 2019). Finally, comparing with the thermal simulation, object of the previous article, it is evidenced that it is quite different, and in the test the temperature reached was approximately 50 ° C higher.

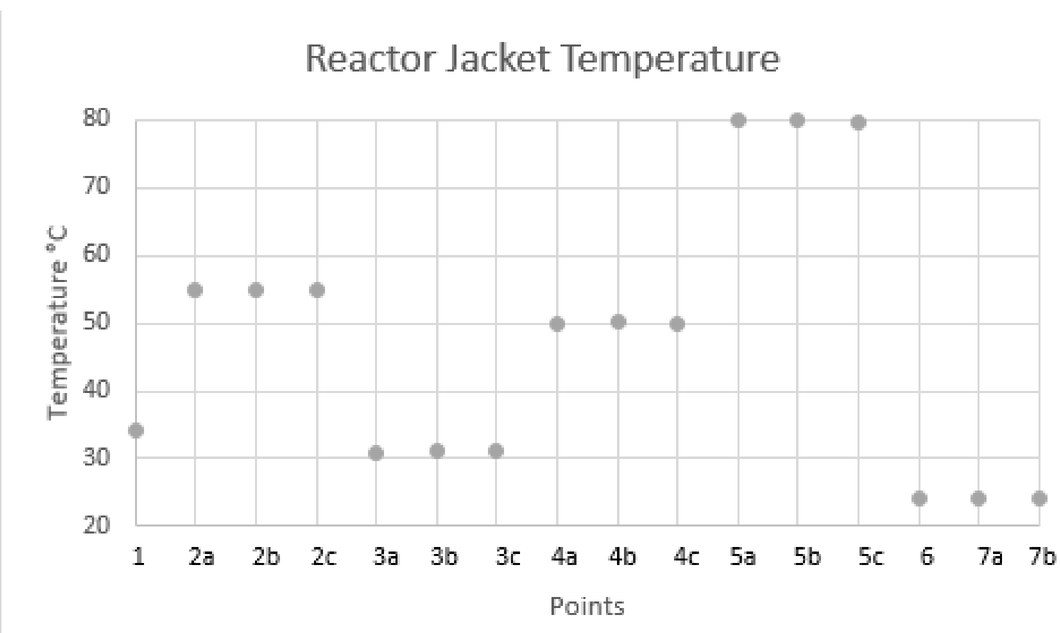

Figure 16. Reactor temperatures upon completion of microwave heating.

4. CONCLUSION.

The material selected to manufacture the body and the top of the reactor, using the Ashby methodology, was PTFE (unfilled), which meets the chemical, mechanical, thermal, and process requirements. In turn, the material selected for the jacket and top was POM-H.

The material index was used in both cases to determine the best material taking into account the best ratio of elasticity limit, density, price, and the different combinations with the materials available in the local market.

The selected materials met the design requirements, there were no plastic deformations in any body due to the pressure developed in the process, surface temperatures of up to 100 ° C were recorded and took no damage whatsoever.

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