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MATERIAL SELECTION FORHIGH PRESSURE REACTOR MICROWAVE-ASSISTED LEACHING OF PLATINUM GROUP METALS

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ABSTRACT

The Colombian Platinum alluvium is 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 reason, this article aims is to select materials for the fabrication of a low-cost industrial reactor with a volume capacity of 180 ml fulfilling the requirements of pressure, temperature, chemical resistance, easy fabrication and safety. Materials selection was made using an Ashby methodology. Reactor was divided in six different sub systems. The first step of the Ashby methodology resulted in a mechanical, chemical and thermal properties list, including objectives to minimized some properties and material index in order to choose the best material. The screen-out step was made using ANSYSGRANTA SELECTOR software and obtained four materials. The availability of this materials in Colombia was constated, choosing finally PTFE and POM-H. A reactor fabrication was made by CNC and a test to measure the reached temperature on the reactor wall was made, resulted in values 20°C below of services temperature of the material.

Keywords: selection materials, ashby methodology; material index.

INTRODUCTION

Platinum is considered a noble metal, that is, resistant to chemical attack [1], it is one of the most no reactive heavy metals, that it is used for medicine [2], as a catalyst in the automobile industry [3], chemical industry, electricity and electronics [4], glass manufacturing, petroleum refining and biomedical. In Colombia, alluvium of platinum groups metals consists oniridium, osmium and palladium [5] but their refining is not carried out since the necessary technology is not available and/or exists a not clear Colombian national mining formalization policy [6], moreover, the extraction and refining 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 reductionetc [7]-[12]. As microwave assisted leaching and microwave assisted synthesis is a efficacy route to synthetic process [13]-[17], then, recovery of platinum, palladium, rhodium and ruthenium [10], [18], [19] 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 [20], [21].

Actually, reactor uses in this microwave process are low capacity (5-10 ml) and a new high-capacity volume reactor is a main objective to dissolution step. The materials selection fulfills design requirements or specifications determined during the design phase isan important point to keep in mind. The settings depend of the requirements design as: 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, but Ashby methodology integrates mechanical factors, physical and chemical properties with ecological ones, and in turn, manufacturing and amalgamation technologies [22], [23], and is supported by an engineering software know as ANSYS-GRANTA SELECTOR makes easier the selection.

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 [22] achieves this objective through 4 steps that are outlined in Figure-1.

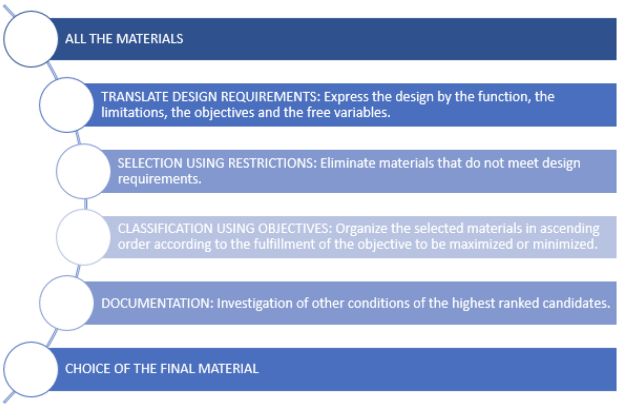


Figure-1. Ashby methodology for the selection of materials.

Translating Design Requirements

At this first stage, the design specifications must be "translated" from the common to the technical language, in terms of specific properties with its quantitative or qualitative values. Translate is maybe, one of the most difficult steps by the mistake on the interpretation of concepts and requirements. The product of the translation is a list with specifics properties required by the design with its numerical value. For this stage, Ashby proposes 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 during its service. Restrictions are design constraints or limitations, which can be static dimensions, specific stresses, specific mechanical settings, chemical or physical properties, etc. These properties don´t describe the purpose of the component, but they are necessary for the design be viable and cannot be negotiated. Objectives are settings or specifications; those that designer want to maximize or minimize. Finally, free variables, are parameters that not affect the design or life in service, but it can affect the purchase decision of the component.

Selection Using Restrictions

This stage, an impartial selection is made take in accounts the different above-mentioned restrictions or limitations. It uses to choose, the entire “universe” of possible materials, or those defined according to the design (Ceramics, polymers, metals, etc.), or those the enterprise provide. The process selection discards materials that do not fill the requirements established in the restrictions, meaning that their attributes are outside the established limits. This can be done easily using the ANSYS EDUPACK software.

Prioritization Using Objectives

On this stage, it is necessary to determine the material index, which is a mathematical expression that relates the property that has been established as function, with ones it wants to maximize or minimize. This materials index can be used on the software to select the best materials that fulfill requirement and objectives. The result of this step is a ranking list in an ascending or descending way, using the value of the objectives-properties.

Documentation

The first selected material of the prioritization list is submitted to a market investigation process to determine the availability of the material in the region, distribution channels, transportation and delivery times, experience and support of the manufacturers. If the first don´t accomplish the expectations, the second one is investigated and if this don´t do, the third, and this cycle is repeated successively until a viable material is found. This type of additional information can often tip the balance towards the choice of a particular material.

Choice of Final Material

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

EXPERIMENTAL

The design of the leaching reactor was carried out by determining the design, functional, and ergonomic parameters. Choosing the best method for calculating the dimensions according to the standards ASME-Division I [24]-[27], BS 5500 Unfired Fusion Welded Pressure Vessel [28]-[30] AD2000 - Merblatter [28], [31], JIS B 8265 [29], [32] and selecting materials using the ANSYS GRANTA SELECTOR software. Leaching medium is a combination of nitric and hydrochloric acid, known as aqua regia, the most efficient ratio is 3: 1 HCl: HNO3 [33]. Maximum working pressure depends on the vapor pressure of the aqua regia, that, it is depending of the maximum working temperatures, that it was reported between 150° C to 250° C[18], [34], [35]. Some commercial reactors have maximum working pressures of 2.4 MPa (350 psi), 5.5 MPa (800 psi) and 8.3 MPa (1200 psi). For this design, take in account the commercial information, and calculate stress by ASME-Division I, the highest design pressure was 10 MPaon the reactor and 50 MPa on the casing [36].

RESULTS

For an economical and easy-to-build design the following model was carried out, it can be seen in Figure-2, that show, six different subsystems to conform the 180 ml sealed reactor, are top of vessel, body vessel, gasket, casing, compression disk and casing top.

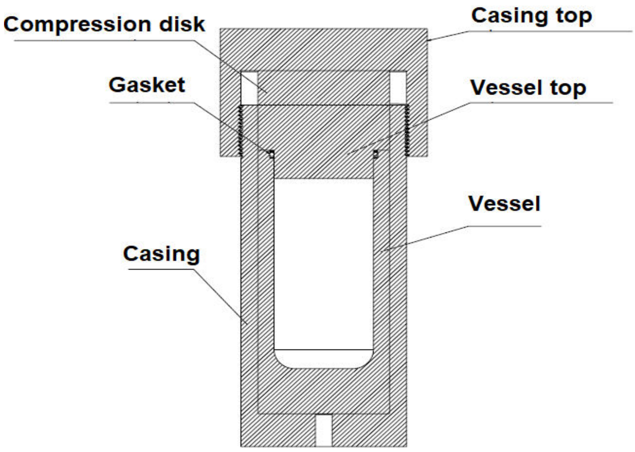


Figure-2. Reactor design.

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 [34], [35]. Also, it possesses high mechanical properties that allow it to withstand up to 1200 psi [34]. Table-1, and Table-2 show the properties and their values necessary to select the material according to the Ashby method, and an extra column named language common, that shows how requirements can be referred from common language. In addition, fabrication properties as 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 [37].

Selection of Materials

Once the properties are defined as specified in previous tables, ANSYS GRANTA SELECTOR software was used entering the values of mechanical, thermal, and chemical properties necessary for the reactor [38]. The screen out process was carried out and the ranking of materials that fulfill both function and restrictions was performed. Finally, the material index was 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.

Vesse ln, Top and Gasket

Four selection materials charts were made according with properties and values of the Table-1 where properties required are listed.

Table-1. Properties for reactor and top vessel and gasket.

	Common Language	Property	Sign	Value (α-#)
Function	Resistant to high pressures.	Yield strength (αf)	≥	10 MPa
Restrictions	Resistant to high temperatures.	Max temp. of service	≥	250 ° C
	Resistant to strongly oxidizing environment	Aqua regia, hydrochloric acid and nitric acid resistance	≥	Excellent.
	Transparent to microwave	Transmissivity	≥	Polymers, ceramics and composites.
	Make it machinable	Machinability	≥	Metals, polymers and compounds
	Do not burn	Inflammability	≥	Non flame
Objective	Economic	Price ($)		Minimize
	Lightweight	Density (ρ)		Minimize
	Do not make an arc with the microwave (security)	Electric conductivity		Polymers and ceramics
Material index	$\frac{\sigma_f}{\rho * \$}$			

The first chart (Figure-3) shows the result of the maximum service temperature ≥250 to 330°C Vs yield strength ≥10 to 50 MPa; second and third charts (Figures 4 and 5) the result of resistance to aqua regia Vs nitric acid and hydrochloric acid Vs flammability, in this step the total materials which fulfill the requirements are reduced only to 4 candidates. Finally, selection material chart (Figure-6) of density Vs price can be appreciated, as well as the search process keeping the lowest values of the aforementioned in mind.

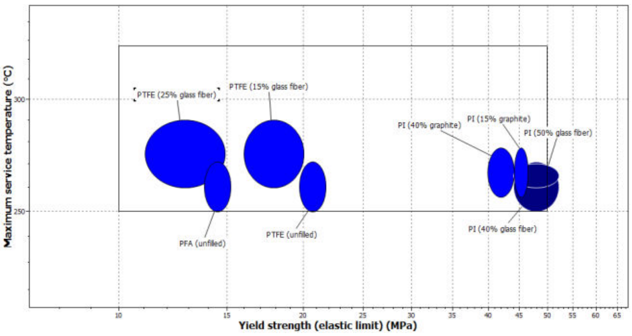
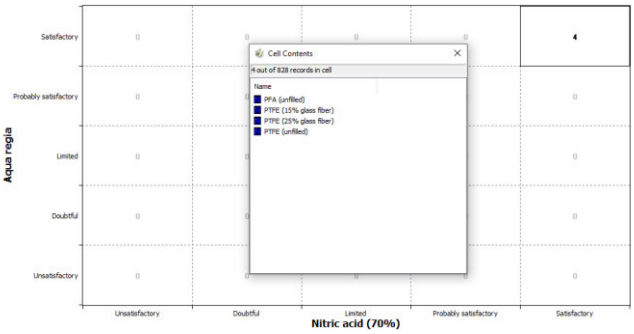


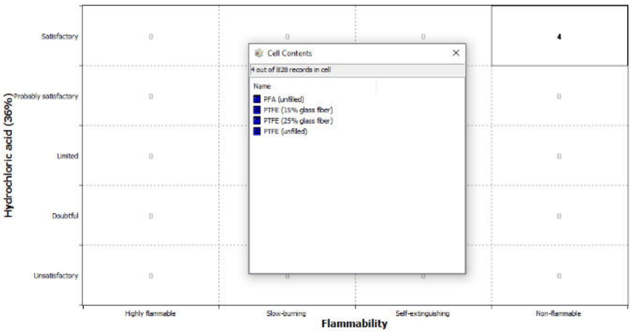
Figure-3. Chart of maximum service temperature Vs. yield strength made by ANSYS GRANTA SELECTOR for body, top and gasket of vessel.



4 out of 828 records in cell

PFA (unfilled)
PTFE (15% glass fiber)
PTFE (25% glass fiber)
PTFE (unfilled)

Figure-4. Chart of Aqua regia Vs. Nitric Acid 70% resistance made by ANSYS GRANTA SELECTOR for body, top and gasket of vessel.



4 out of 828 records in cell

PFA (unfilled)
PTFE (15% glass fiber)
PTFE (25% glass fiber)
PTFE (unfilled)

Figure-5. Chart of hydrochloric acid Vs. flammability made by ANSYS GRANTA SELECTOR for body, top and gasket of vessel.

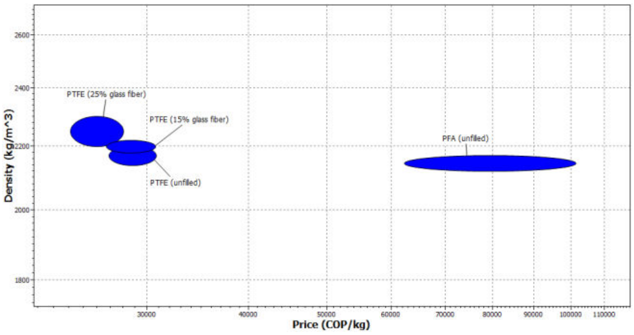


Figure-6. Chart density Vs. pricemade by ANSYS GRANTA SELECTOR for body, top and gasket of vessel.

The resulting materials in the selection using restriction are:

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

The material index allows the maximization of the functional property required by the design, being, in this case, the yield strength and the minimization of cost and density properties (Figure-7). The material index can be drawn as a lineal curve in a chart Yield strength Vs Density*Cost with a slop that is the inverse (1/x) of the exponent number of the function property, in this case the exponent of yield strength is 1, then, slope is also. This line allows to find the best ratio of the highest technical property and the lowest price and density.

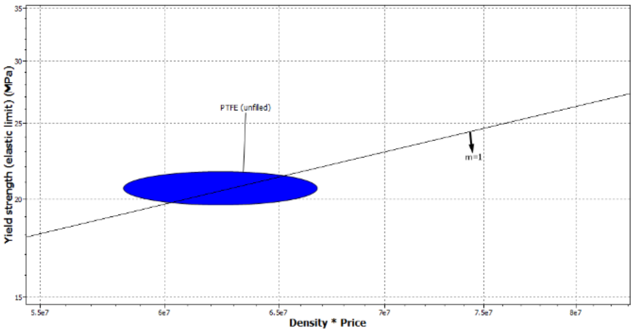


Figure-7. Chart yield strength Vs. density * price made by ANSYS GRANTA SELECTOR for body, top and gasket of vessel.

Complying with the mechanical, thermal, and chemical properties and also using the material index defined for this application (Table-1), the PTFE (unfilled) (Figure-7) was selected for the construction of the reactor, its top and gasket, this material is commonly called Teflon and it is considered a thermosetting polymer [39] [40] [41]. This material is easily available in Colombia.

Casing, Top Casing and Compression Disk

For those three subsystems, 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 and shear are essential to the casing and top parts, and compressive strength to the compression disk. Required properties and their values are listed in Table-2.

Anew four selection materials charts were made according with properties and values of the Table-2. The first chart (Figure-8) shows compression resistance Vs. yield strength since 50 MPa; Figure-9 shows maximum service temperature vs. tensile strength chart with values higher than 90 and 50 to 120 MPa respectively, lastly; Figure-10 shows density and price chart require to minimization.

Table-2. Properties for casing, top and compression disk.

	Common Language	**Property**	**Sign**	**Value (α-#)**
Function	**Resistant to high pressures**	**Yield strength (αf)**	**≥**	**50 MPa**
Restrictions	Resistant to high pressures	Compressive strength (αc)	≥	50 MPa
	Transparent to microwave	Transmissivity	≥	Polymers, ceramics and composites
	Make it machinable	Machinability	≥	Metals, polymers and compounds
	High temperature resistance	Maximum service temperature	≥	90°C
	Do not make an arc with the microwave (security)	Inflammability	≥	Non flame
Objective	Economic	Price		Minimize
	Lightweight	Density (ρ)		Minimize
	That does not arch with microwaves.	Electric conductivity.		Minimize
Material index	$\frac{\sigma_f}{\rho * \$}$			

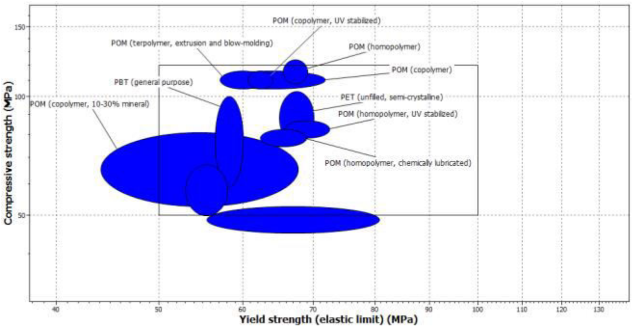


Figure-8. Chart compressive strength Vs. yield strengthmade by ANSYS Granta Selector for casing, top and compression disk.

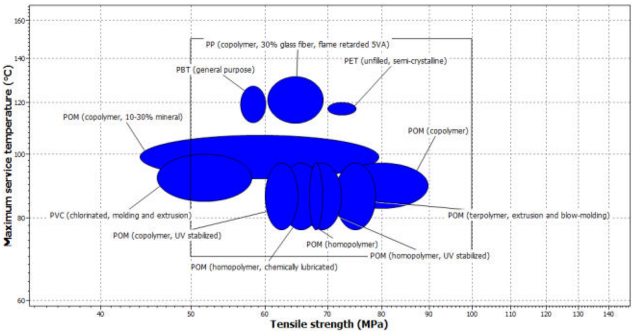


Figure-9. Chart maximum service temperature Vs. tensile strengthmade by ANSYS GRANTA SELECTOR for casing, top and compression disk.

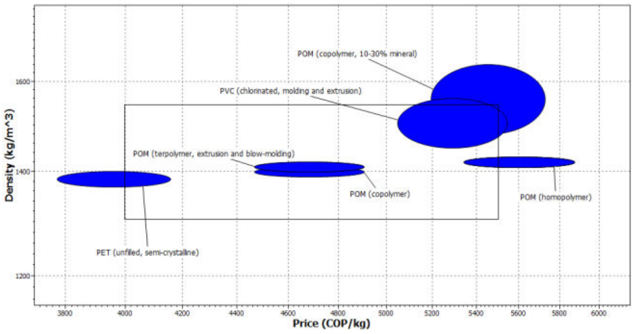


Figure-10. Chart density Vs. pricefor casingmade by ANSYS GRANTA SELECTOR, top and compression disk.

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 strength and minimize the density and price. In Figure-11 it can see the linear curve with a slope of 1, chosen accordingly to this index as before was explained.

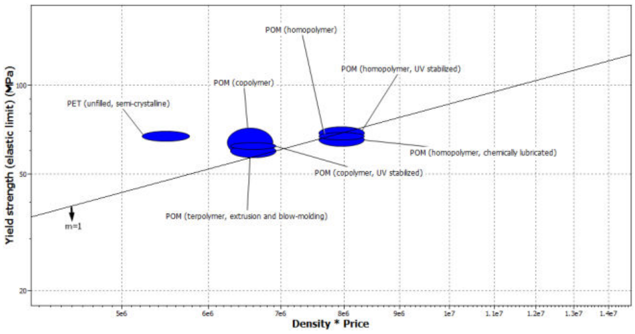


Figure-11. Body and shirt top. Yield strength vs. density * Pricemade by ANSYS GRANTA SELECTOR.

The material selected for the liner index material, is POM Terpolymer, follow for POM Homopolymer in its three version (homopolymer; chemical lubricated and UV stabilized). Documentation, the final step of the Ashby methodology (Figure-1) determined the easiest one to acquire in Colombia was POM-Homopolymer. This is a material that fulfill mechanical, thermal and chemical properties [42] [43].

Manufacture of 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 casing 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 [44]. Figures 12 to 14 show the different reactor components.

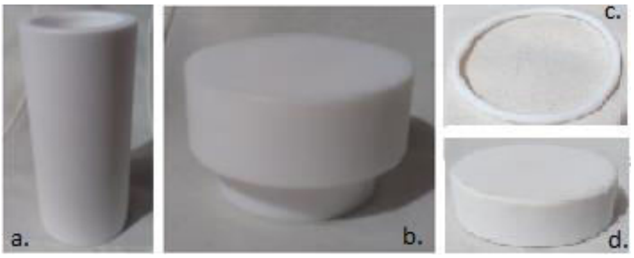


Figure-12. Reactor a. Body, b. Top, c. Gasket, d. Compression disk.

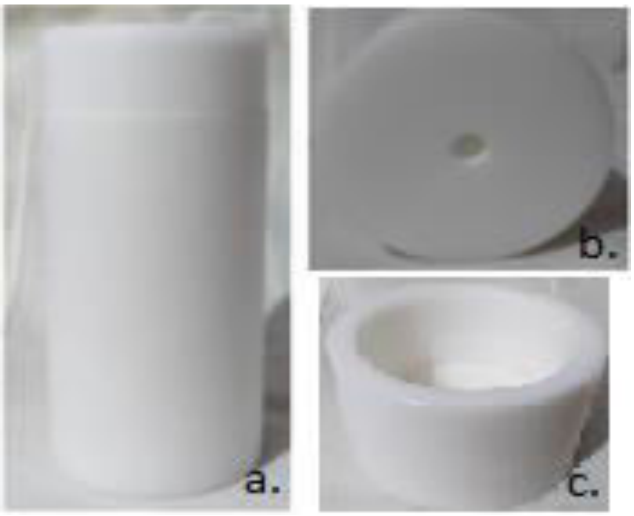


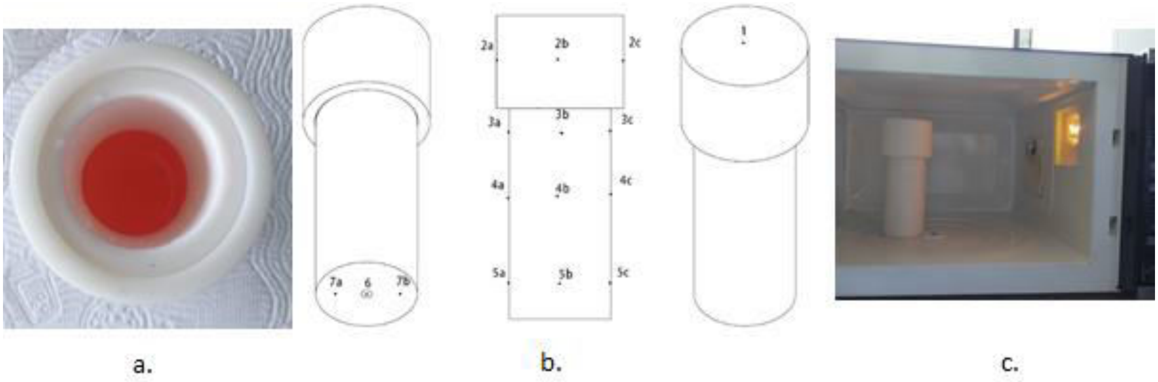
Figure-13. Casinga.- Body, b.-Compression disk, c.-Top.

Figure-14. Reactor vessel.

Reactor Test on Microwave Oven

Test for the reactor was carried outona Samsung Microwave, 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.

In Figure-16 the temperatures reached in the casing wall 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 [42].



a.

b.

c.

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

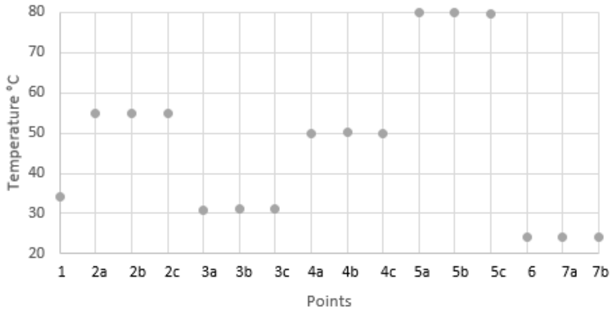


Figure-16. Casing temperatures in two minutes of heating in microwave oven.

CONCLUSIONS

The material selected to manufacture the vessel, top and gasket of the reactor, using the Ashby methodology, was PTFE (unfilled), which fulfill the chemical, mechanical, thermal, and process requirements. In turn, the material selected for the casing, top and compressive disk was POM-H.

The material index was used in both cases to determine the best material taking into account the best ratio of yield strength, density and 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 80° C were recorded and took no damage whatsoever.

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