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Design and construction of a batch reactor with external recirculation to obtain biodisel from residual oil frying under subcritical conditions

Diseño y construcción de un reactor discontinuo con recirculación externa para obtener biodiésel a partir de aceite de fritura en condiciones subcríticas

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Abstract

A batch reactor was designed and built to obtain biodiesel from frying oil under sub-critical conditions, with the purpose of reducing the reaction time to the minimum possible. The design process is focused on the selection of the material and the verification of its resistance by means of a FEM analysis from a Design of Experiments (DOE). Three levels of pressure, temperature and wall thickness, respectively, and a material categorical factor at two levels were considered. The results obtained were that the appropriate material for manufacturing the reactor is 304 stainless steel with a design safety factor of 1. For constructing the system it was also necessary to select all the complementary components. The final operation tests showed that it is possible to safely obtain the biofuel in the batch reactor with a degree of conversion 88%, in a range of 5 to 8 minutes.

Keywords: Biodiesel, Discontinuous reactor, subcritical conditions, DOE, Finite elements.

Resumen

Se diseña y construye un reactor discontinuo para obtener biodiésel a partir de aceite de fritura en condiciones subcríticas con la intención de reducir el tiempo de reacción al mínimo posible. El proceso de diseño se centra en la selección del material y la verificación de su resistencia mediante un análisis FEM a partir de un diseño experimental DOE. Se consideran tres niveles de presión, temperatura y espesor de pared, respectivamente, y un factor categórico material a dos niveles. Los resultados obtenidos permiten determinar que el material apropiado para la manufactura del reactor es acero inoxidable 304 con un factor de seguridad de diseño de 1. Para el proceso de construcción del sistema es necesario también la selección de todos los componentes complementarios. Las pruebas finales de funcionamiento muestran que es posible obtener el biocombustible en el reactor discontinuo con un grado de conversión del 88 % de manera segura en un rango de 5 a 8 minutos.

Palabras clave: biodiésel, reactor discontinuo, condiciones subcríticas, DOE, elementos finitos

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1. Introduction

Most countries worldwide are concerned about reducing the emission of greenhouse gases, incorporating new sources of energy as an alternative to fossil fuels and recovering deforested territory with arable and non-arable vegetation [1]. For this purpose, the production of biofuels is a tangible alternative [2–4]. It is expected that increasing the production of biofuels will not only contribute to the conservation of the environment, but also to the economical and social development of the producing countries [5].

Conventional techniques for producing biodiesels use reactors. A chemical reactor is a complex device in which heat and mass transfer, diffusion and friction may occur together with the chemical reaction under control and safety devices. There are different types of reactors according to the form of operation, the type of internal flow and the phases they house [6–9]. In general, it is sought to determine the size and type of reactor, as well as the operation method necessary to obtain the final product [10].

One of the important parameters intended to be improved is the time for obtaining the biodiesel. Times between 20 and 180 minutes have been reported in previous studies [11]; in particular, in residual cooking oils this time is 90 minutes.

In recent years, attention has been focused on seeking raw materials different than vegetable oils such as soy, palm, etc. This is mainly due to the cost of obtaining the raw material, which is approximately 70 % of the total cost of obtaining biodiesel [6–8]. The frying oil is another source of raw material, whose advantage over other raw materials is that they are in the category of residuals. This feature gives it a great viability because it contributes to the reduction of the environmental pollution.

In Latin America, the residual frying oil has the potential to be used as a primary source for producing biodiesel [12]. There is a large industry of fast and traditional food that uses frying oil, which becomes a waste after being used. For example, in Colombia 35 % of the yearly production of 162 million liters of cooking oil becomes a residual that is discarded in sewers [13], and Spain produces 150 million liters of used vegetable oil yearly [14].

On the other hand, in the industrial, educational and residential sectors, there are countless products and systems developed to accomplish a specific task. All of them employ engineering materials chosen to achieve an optimal performance. The process for obtaining a product comprises three macro-processes: design, manufacturing and operation tests. The design stage starts determining the requirements and constraints to further carry out a material selection process [15].

In recent years, two methods for material selection have been used in engineering development: traditional and graphical. The former is based on the knowledge and experience of the engineer, the latter on graphical maps of engineering materials organized according to their physical and mechanical properties [16].

The graphical method was developed as a very important support in the stage of conception and development of the product [17, 18]. The graphical method from Michael Ashby has been the most useful one in recent years. It is a methodology that acts as a guide for material selection, considering the attributes that are related with each other by means of graphical selection tables [18, 19]. Performance indices are utilized, which refer to groups of material properties with the purpose of maximizing or minimizing them according to the specific requirement. They are derived from the objective function of the system, and are expressed by means of mathematical equations.

Various authors have reported the use of the Finite Element Analysis (FEA) technique for designing and verifying chemical reactors subject to multiphase modeling [20–23]. Studies, such as [24–26], mention that parametric design provides mechanical systems with the capability of synthesizing, simplifying and economizing the design process, which enables amplifying and exploring possibilities of solution.

In the current study, it was carried out the design and construction of a batch reactor with external recirculation to obtain biodiesel from residual frying oil under subcritical conditions, with the purpose of reducing the production time within a controlled and safe framework that fulfills engineering requirements. To this effect, it was proceeded to select the materials and verify the resistance under the stress regime using the finite elements technique.

2. Materials and methods

The following methodology was applied: selection of materials, design by means of finite element analysis (FEA), construction of the system and operation tests. It is remarked the fact that the design of the reactor was initiated with the observation of previous designs, to come up with a completely new design which enables reducing the time to obtain the biodiesel.

2.1. Selection of materials

It was utilized the graphical method from Ashby, with the aid of the CES EDUPACK educational software. It was defined the required functionality, the objective and the constraints of the reactor (Table 1). From Hooke´s law which gives the tensile stress in the elastic zone (σ) as the product of Young´s modulus (E) times the strain (ϵ), and the formula of mass (m) as a function of volume and density (ρ), it was determined the performance index (Equation 1).

Table 1. Definition of the design requirements

Definition	Detail
Functionality	House the thermo-chemical process
Objective	Minimize mass of the reactor (ρ) and maximize mechanical resistance (σ)
Constraints	Withstand minimum and maximum temperatures of 160 °C and 400 °C, respectively
	Withstand minimum and maximum pressures of 250 psi and 3000 psi, respectively
	Resistance to oxidative and corrosive processe
	Low cost
	Good machinability and solderable

$$m = \frac{F}{\epsilon \cdot E} \cdot L \cdot \rho = F \cdot L \cdot \left[\frac{\rho}{\sigma}\right]$$
$$\Rightarrow Performance\_indexño = \frac{\rho}{\sigma} \quad (1)$$

Taking all the functionalities and initial constraints for the reactor, it was obtained the possibility of 600 alternatives of material among a total of 3900 options. The adjustment of the selection was carried out according to the specific conditions of service (high mechanical resistance, good performance at high temperatures and resistant to chemical elements), local availability of the material at the smallest cost possible and a high density which enables the moving stability of the system. (Figure 1).

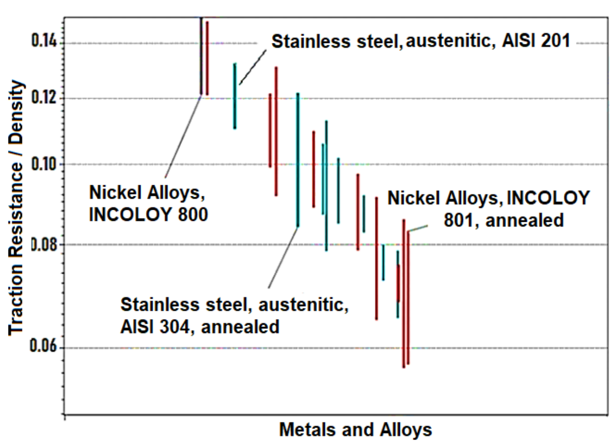


Figure 1. Plot stress-density for selection of materials [18]

The input variables were: a maximum price of 10 USD, density between 5000 and 8000 kg/m^3, service temperature of 500 °C, resistance to alkaline substances and substances with pH greater than 7, molding and machinability capabilities that result in 18 possibilities of material framed in three groups: austenitic stainless steels, annealed austenitic stainless steels and nickel alloys.

2.2. Finite elements analysis (FEA)

Once the selection of materials for the reactor has been performed, it was chosen the AISI 304 steel and the nickel alloy to enter the FEA experimental design. On the other hand, the dimensions of the batch reactor were stated such that it yields a continuous recirculation capability of 1 liter.

A complete factorial design was stated where it was taken into account the three main factors (constraints/requirements) of the reactor; pressure, temperature and wall thickness in three levels, respectively. A categorical variable (material) in two levels was also considered. Table 2 shows the experimental design. The ranges of pressure and temperature values were based on studies regarding obtaining biodiesel in subcritical and supercritical conditions.

Table 2. Design of experiment

Factors	Levels		
	Bajo	Medio	Alto
Pressure (Mpa)	1,72	4,83	20,7
Temperature (°C)	160	200	400
Wall thickness (mm)	1,5	2	3
Material	AISI 504 steel	Allow Nickel	-

It was performed a thermomechanical analysis by means of finite elements using a multi-parametric symmetric 2D model created in Ansys APDL, to evaluate the mechanical resistance of the reactor to the internal conditions of pressure and temperature specified in the design of the experiments. It was used a quarter of the cross section due to the two planes of symmetry existing in the model, Figure 2. For the two materials used in this study it was defined an elastic linear isotropic model of the material, whose properties are shown in Table 3.

Table 3. Mechanical and thermal properties for the linear elastic isotropic model of the material

Mechanical and thermal properties	Material	
	AISI 304	Ni 800
Elasticity modulus (GPa)	193	196
Poisson coefficient	0,3	0,34
Yield stress (MPa)	220	335
Density (kg/m^3)	7850	7940
Service temperature (°C)	850	816
Conductivity (W / kg-K)	16,2	11,5
Specific heat (J/kg-°C)	500	460
Thermal expansion coefficient (x 10^6 °C^{-1})	17	12

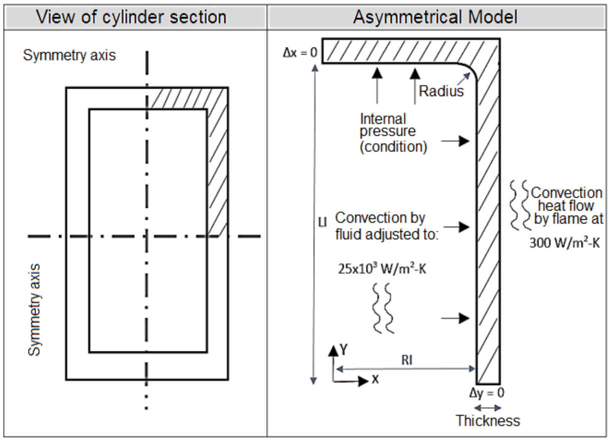


Figure 2. Geometry and limiting conditions of a model of asymmetrical reactor of quarter of axis for a thermomechanical analysis

The model was discretized with PLANE182 quadrilateral elements in their axisymmetric version; for all cases, the size of the element was established at 1/5 of the cylinder wall thickness, because this value assured a convergence smaller than 5%. The model has 1830 nodes and 1520 elements. The limiting conditions for the stationary thermomechanical analysis are shown in Figure 2.

Two types of finite elements analysis were carried out: one thermal stationary with the temperature and convection conditions specified to obtain displacements and thermal strains, and then a static structural analysis with the boundary conditions indicated such that both studies were coupled in a multi-physical analysis. The validation of the finite elements model of the first type of analysis was carried out by means of the Thin Wall Pressure Vessel theory (TWPVt), and the circumferential or tangential (SZ) and longitudinal or axial (SY) stresses were evaluated, because this theory does not take into account the concentration of stresses, such as covers, holes or abrupt changes in the cross section. In the second finite element analysis, the influence of pressure and temperature was evaluated, and the results of the equivalent Von Mises stress were compared to the yield stress of each material to determine the safety factor of the reactor.

$$\sigma_e = \sqrt{(\sigma_x + \sigma_y) - 3(\sigma_x \sigma_y - \tau_{xy}^2)} \tag{2}$$

where:

σ_e=Von Mises stress
σ_x=Stress normal to the X axis
σ_y=Stress normal to the Y axis
τ_{xy}^2=Shear stress in the XY plane

According to the theory of thin wall pressure vessels (internal diameter/thickness ratio greater than or equal to 10), there are two main stresses which are a function of the internal pressure (p), internal radius (r) and wall thickness (t). These are longitudinal or axial and circumferential or tangential stresses, whose calculation formulas are (3) and (4), respectively:

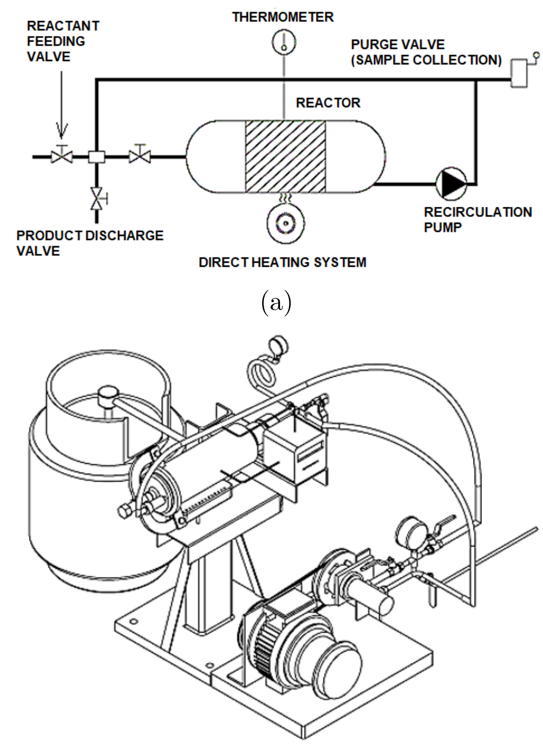
Longitudinal or axial stress (SY):

$$\sigma_1 = \frac{pr}{2t} \tag{3}$$

Circumferential or tangential stress (SZ):

$$\sigma_t = \frac{pr}{t} \tag{4}$$

2.3. Construction of the system



(a)

(b)

Figure 3. Batch reactor a) System scheme, b) 3D configuration

The batch reactor (Figure 3) is constituted by a tank, a circulation tube, and a recirculation pump to stir the reagents. The tank consists of a cylindrical tube with a length of 280 mm, an internal diameter of 71 mm and a wall thickness according to the minimum thickness resulting from the design (FEM analysis). The tank is coupled to a tube of length 1000 mm and diameter of 12 mm and a high-pressure pump that rotates at 250 rpm for recirculation and steering. As the tube, the reactor will be manufactured using the appropriate material studied in section 2.2 that is available at the lowest cost. The loading flow (3 l/min) is driven by a Motovario TXF005 motor. A thermocouple connected to the temperature controller and a pressure gauge to measure the internal pressure of the vessel, were place on the reactor cover. The cover comprises a flange with a teflon gasket. Two Problock pressure gauges, from 0 to 150 psi, were placed on the upper part of the reactor. The temperature of the reactor is recorded by a Syscon AKC CB100 temperature controller.

2.4. Operational tests

The tests were carried out at temperatures of 160, 180 and 200 °C, with methanol-oil molar ratios of 6:1 and 9:1, catalyst percentage (NaOH) of 0.5 and 1 % and for a reaction time of 5 to 10 minutes to verify the functionality of the batch reactor with continuous recirculation capacity of 1 liter, reaching pressures of 250 psi (subcritical condition). For obtaining crude biodiesel, it will be subject to distillation and wash with atomized water until the wash water comes out clean. After eliminating the secondary products, the biodiesel will be heated at 105 °C for ten minutes to dry or eliminate the water and the methanol residues. After cooling, a volume of refined biodiesel was obtained.

3. Results and discussion

3.1. Numerical and analytical results

The results of the static structural analysis are shown in Table 4. The AISI 304 stainless steel and the Ni 800 nickel alloy have very similar elasticity module and Poisson ratio, which indicates that the results of the longitudinal (SY) and circumferential (SZ) stresses practically do not vary, it only changes the safety factor, defined as the ratio between the yield stress of the material and the equivalent maximum stress achieved. It is important to take into account that, in this analysis, the safety factor was only obtained with the circumferential and longitudinal stresses, which does not necessarily represent a complete stress condition of the point under analysis. According to the results of the finite elements model, it satisfactorily agrees with the theory of the thin wall pressure vessels. The maximum error was approximately 4.2%.

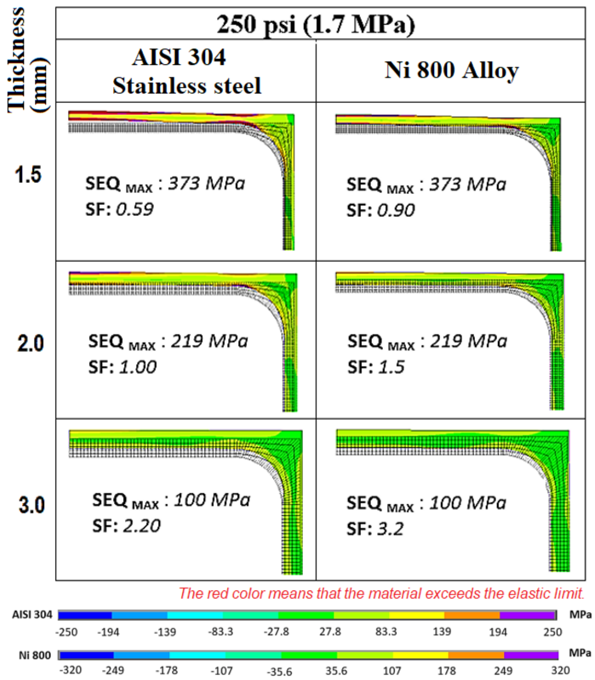
3.2. Influence of the internal pressure and temperature of the reactor

The study of the thermomechanical coupling of a quarter of the cross section of the reactor was utilized to determine the influence of the internal pressure and the temperature. Tables 5, 6 and 7 show the influence of the internal pressure for a condition of constant temperature at 160 °C. The color legend was separated for the AISI 304 stainless steel and for the Ni 800 nickel alloy, since they have a different elastic limit. The red color in Table 4 means that the material exceeds the elastic limit and there is the possibility of permanent deformations that lead to the failure of the bioreactor. The deformation scale with respect to the not deformed condition is the same for each case.

Table 4. Comparison of the longitudinal σ_l (SY) and circumferential σ_t (SZ) stresses from the finite element analysis (FEA) and the thin wall pressure vessels theory (TWPVt)

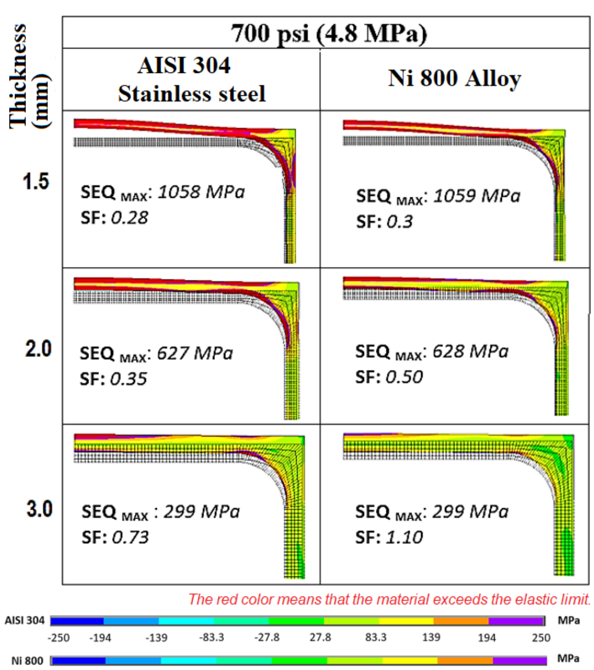
Exp	Pressure (psi)	DOE Thickness (mm)	Material	FEA SY	TWPVt σ_l	Error %	Safety factor	FEA SZ	TWPVt σ_t	Error %	Safety factor
1	250	1,5	AISI 304 / Ni 800	2857	2917	2,1	10,5/15,8	5960	5834	2,1	5,0/7,6
2	250	2	AISI 304 / Ni 800	2127	2188	2,8	14,1/21,2	4502	4375	2,8	6,7/10,0
3	250	3	AISI 304 / Ni 800	1399	1458	4,1	21,4/32,2	3045	2917	4,2	9,9/14,8
4	700	1,5	AISI 304 / Ni 800	8066	8236	2,1	3,7/5,6	16827	16472	2,1	1,8/2,7
5	700	2	AISI 304 / Ni 800	6003	6177	2,8	5,0/7,5	12711	12354	2,8	2,4/3,5
6	700	3	AISI 304 / Ni 800	3951	4118	4	7,6/11,4	8597	8236	4,2	3,5/5,2
7	3000	1,5	AISI 304 / Ni 800	34614	35346	2,1	0,9/1,3	72216	70692	2,1	0,4/0,6
8	3000	2	AISI 304 / Ni 800	25784	26510	2,7	1,2/1,7	54552	53019	2,8	0,5/0,8
9	3000	3	AISI 304 / Ni 800	16956	17673	4,1	1,8/2,7	36898	35346	4,2	0,8/1,2

Table 5. Numerical analysis of the influence of the internal pressure of the reactor



Constant temperature = 160 °C and internal pressure 250 psi (1.7 MPa)

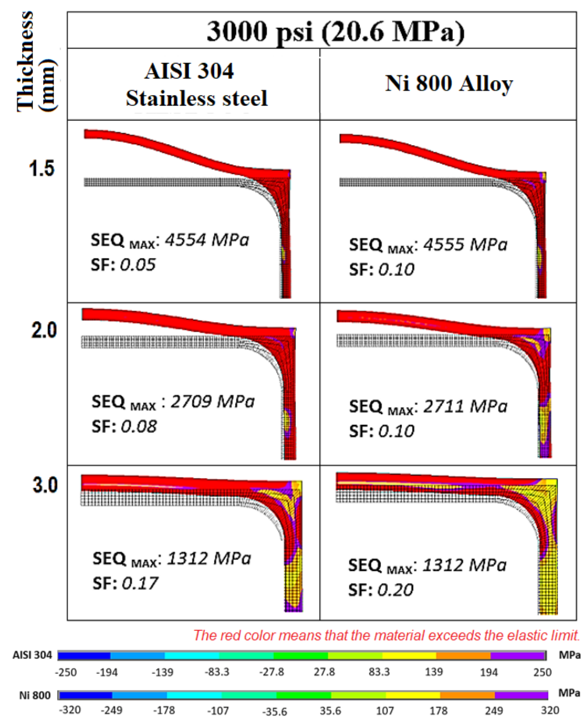
Table 6. Numerical analysis of the influence of the internal pressure of the reactor



Constant temperature = 160 °C and internal pressure 700 psi (4.8 MPa).

The finite element analysis shows similar results of equivalent Von Mises stress for both materials, because the elasticity modules and Poisson ratios are very similar; however, the safety factors are different because there is a difference of about 100 MPa between their elasticity limits. The achieved maximum Von Mises stress was larger for thicknesses of 1.5 mm of the reactor wall, a minimum for thicknesses of 3 mm. For an internal pressure of 250, 700 and 3000 psi, the maximum Von Mises stress was 373, 1058 and 4555 MPa, respectively.

Table 7. Numerical analysis of the influence of the internal pressure of the reactor



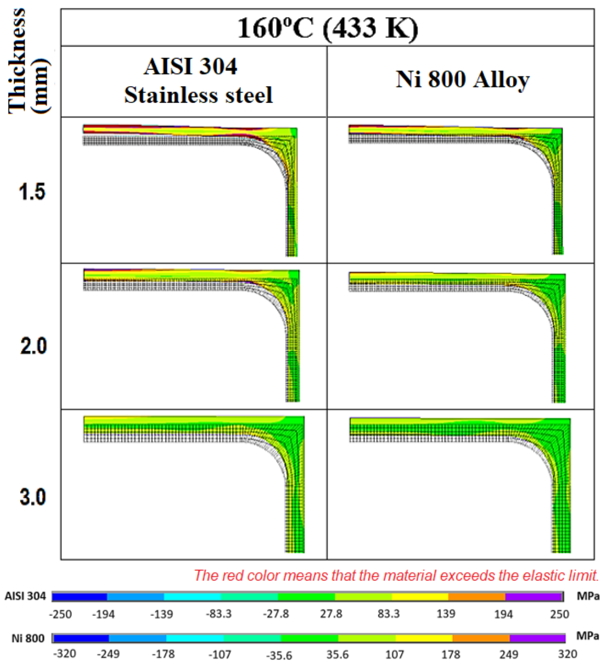
Constant temperature = 160 °C and internal pressure 3000 psi (20.6 MPa)

With the conditions of the axisymmetric finite elements modeling developed in this study, it is verified that for the case of the AISI 304 stainless steel the design is safe only for subcritical conditions of pressure up to 250 psi and a minimum thickness of 2 mm, for which a safety factor of 1.00 was obtained. The safety factor is calculated as the ratio between ratio between the elastic stress of the material and the equivalent maximum stress achieved.

For the Ni 800 nickel alloy, the internal pressure may be increased up to 700 psi with a thickness of 3 mm, where the safety factor is 1.10. For conditions of supercritical internal pressure (3000 psi), the safety factors are below 1.00 in all cases, which indicates that the material has a very high probability of failure, For this condition, the thickness of the reactor wall must be drastically increased.

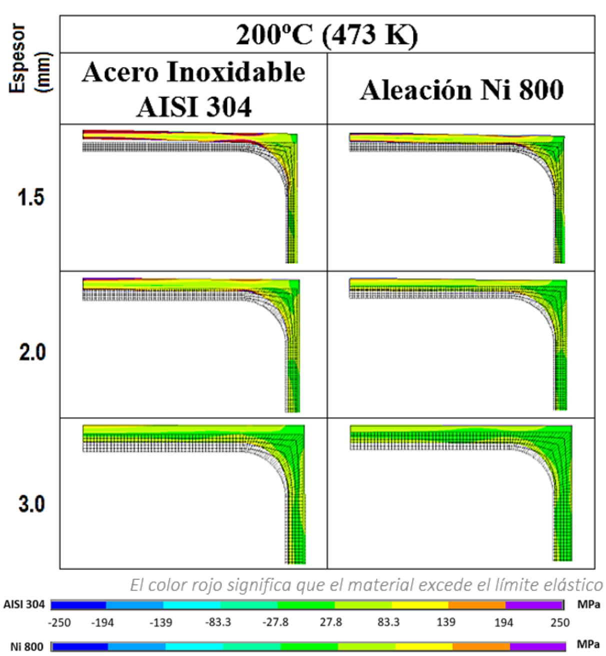
Tables 8, 9 and 10 show the influence of the temperature for a condition of constant internal pressure at 250 psi.

Table 8. Analysis of the influence of the temperature of the reactor



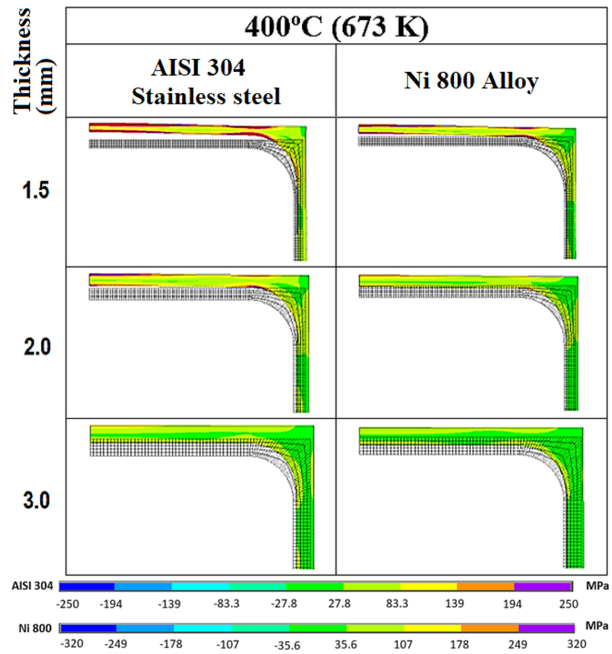
Constant pressure = 250 psi and temperature of 160 °C

Table 9. Analysis of the influence of the temperature of the reactor



Constant pressure = 250 psi and temperature of 200 °C

Table 10. Analysis of the influence of the temperature of the reactor



Constant pressure = 250 psi and temperature of 400 °C

For both materials, it may be verified that the equivalent maximum stresses have a minimum variation as the temperature increases to values of 160, 200 and 400 °C; therefore, in this model the results are only influenced by the change in internal pressure. It is assumed that this is due to two factors: the temperature gradient is minimum at the reactor wall due to the stable condition that is reached with the internal thermal fluid (biodiesel) and the external convective flow (fire). On the other hand, none of the temperatures reached exceeds the service temperatures of the materials, 850 °C for the AISI 304 stainless steel and 816 °C for the Ni 800 nickel alloy. Therefore, in a further research it is important to develop a fatigue analysis to estimate the useful life of the reactor.

3.3. Pruebas de funcionamiento

With the prototype developed according to section 2.3 and the considerations of the numerical analysis (FEM), it was proceeded to conduct the operational tests in the lab.

After executing the procedure stated in the methodology, it was obtained a volume of refined biodiesel of transparent yellow color. The densities of biodiesel obtained were analyzed (smaller density is better), taking as reference temperatures of 160 °C, 180 °C and 200 °C used for the transesterification (Figure 4). When comparing the means of the biodiesel results, statistically significant differences were found; the largest density occurred for 200 °C (0.89 g/ml), while at 160

°C and 180 °C the density was 0.88 g/ml. The time for obtaining the biodiesel starts from 5 to 10 minutes, being stable to obtain it after 8 minutes.

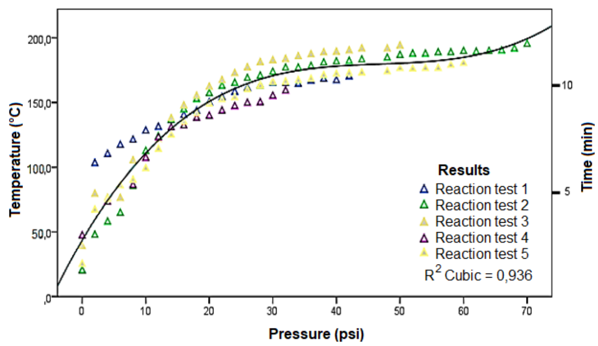


Figure 4. Behavior of the internal reaction of the reactor as a function of the temperature, pressure and time

4. Conclusions

The graphical method of material selection enables reducing the time for selecting the material. The great number of options for manufacturing a reactor makes essential the need for a refinement in the selection when determining the service conditions, such as temperature gradients, pressure ranges, chemical influences, pH, and considerations such as cost and availability of the material.

It was performed a symmetric finite element multiparametric analysis in 2D axis using the Ansys APDL software, to evaluate the mechanical resistance of the reactor. Such analysis enables determining the influence of the variables in the thermomechanical behavior of the reactor. Results show that the design is safe only for subcritical conditions of pressure up to 250 psi and a minimum thickness of 2 mm for the case of the AISI 304 stainless steel, where a safety factor of 1.00 was obtained. In the case of the Ni 800 nickel alloy, the internal pressure may be increased up to 700 psi with a minimum thickness of 3 mm, where the safety factor is 1.10. At the limiting conditions of this model, the temperature does not have influence on the Von Mises equivalent stress.

A volume of 38.48 cm^3 was obtained as a function of the diameter and length of the tubular section, which represents the 3,47% of the total volume of the reactor, thus, it was considered insignificant. On the other hand, the recirculation flow managed by the pump during the operation of the reactor was 3 l/min; as a consequence, the fluid that passes through the pipe has a residence time (cylindrical section / pump flow) equal to 0.026 min (1.6 seconds), which makes evident that the effect of the behavior of the piston flow in the tubular section is not significant with respect to the reaction process that occurred in the tank of the reactor.

Through the reaction process, it was observed that, as temperature increases, more soap is formed, which increases the biodiesel density. However, with respect to the biodiesel density obtained in subcritical conditions, these values are within the European standard that establishes a minimum range of 0.86 g/ml and a maximum of 0.90 g/ml. This biodiesel quality was achieved in an approximate time of 5-8 minutes.

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