ISSN 0101-2061 (Print)
ISSN 1678-457X (Online)

Food Science and Technology

DOI: https://doi.org/10.1590/fst.05118

Appliance of a high pressure semi-batch reactor: supercritical transesterification of soybean oil using methanol

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Abstract

This work presents the results of a supercritical transesterification process of soybean oil using methanol, performed in a laboratory high pressure semi-batch reactor. A full description of the equipment and a standard operating procedure are also provided. Since fossil fuels are not renewable, biofuels such biodiesel, have gained attention over the past decades. Biodiesel is the result of the transesterification of triacylglycerols in vegetable oils or animal fats and it works as an alternative fuel for compression-ignition engines. Methanol was chosen to perform the reaction, at molar ratios of 1:24 (oil: alcohol) and 0.1 (CO_2:methanol), temperature of 280 °C and pressure of 130 bar. In order to decrease the reaction time, CO_2 in the supercritical state was used as cosolvent. The yield of biodiesel produced was equal to 95.19% in only ten minutes of reaction. A high yield of methyl esters (84.41%) was detected by gas chromatography-mass spectrometry. In general, 10 compounds were identified. The component in higher concentration was linoleate, followed by methyl hexadecanoate, oleic acid methyl ester and methyl stearate. The ester conversion rate means that the process with supercritical methanol can be successfully applied to produce biodiesel.

Keywords: autoclave; transesterification; high-pressure reaction; supercritical conditions.

Practical Application: The development of new techniques is of great importance for the future generations, since the more the world population grows, the more energy is required to maintain life quality. This includes the production of biofuels, considering that fossil fuels are not renewable. Thus, to produce biodiesel, the supercritical transesterification corresponds to an alternative to the standard transesterification method since it does not require the use of conventional catalysts, generates less undesirable products, with a high rate of alkyl esters, in a short-time reaction.

1 Introduction

Nowadays, the need for renewable energy sources increases everyday due to the greater demand for petroleum products, which are not renewable and tends to be more expensive in the future. Another aspect of these products is that they produce a lot of harmful composites, such as carbon monoxide, nitrogen and sulfur oxides. Therefore, attention has been focused on developing renewable, biodegradable or alternative fuels, such as biodiesel to replace petroleum based fuels (Fukuda et al., 2001; Gopinath et al., 2009; Javidialesaadi & Raeissi, 2013).

Biodiesel is the result of the transesterification of triacylglycerols in vegetable oils or animal fats. It works as an alternative fuel for compression-ignition engines. Since the transesterification reaction needs a short-chain alcohol, methanol and ethanol are the most used (Bunyakiat et al., 2006; Ali et al., 2013).

At first, the transesterification is a reversible reaction (Figure 1). However, the glycerol formed is immiscible in biodiesel, greatly reducing the extent of the reverse reaction. The miscibility of glycerol in the biodiesel is a factor favoring the yield of reaction; however, the possibility of forming stable emulsions, in certain cases, may require a long period of rest for phase separation of biodiesel and glycerol (Atadashi et al., 2011; Lourenço & Stradiotto, 2009).

In biodiesel production with base catalysts, besides the transesterification reactions, saponification of alkyl esters or of triacylglycerols themselves may also occur. Saponification is the formation of soap, i.e., salts of fatty acids, which can also occur due to a high content of free fatty acids in oils or fats. The soap formation involves three problems: it consumes the catalyst used in the reaction; it causes an increase in viscosity or formation of gels that interferes in the reaction. Also, the presence of soap in the reaction mixture stabilizes the biodiesel/glycerol emulsion. A technical problem is also related to the use of biodiesel with high soap content: engine damage due to fouling and corrosion (Van Gerpen, 2005; Ma & Hanna, 1999; Meher et al., 2006).

Received 15 Mar., 2018
Accepted 26 Nov., 2018

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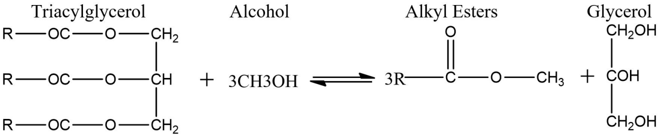

Figure 1. Overall scheme of a generic triacylglycerol transesterification with methanol.

The biodiesel production may involve the use of catalysts: acids but mostly bases (hydroxide or sodium alkoxide or potassium). It has the advantage of being carried out at room temperature and still be faster than the transesterification catalyzed by acids, which needs to be performed at elevated temperatures (170 °C) (Rinaldi et al., 2007; Vicente et al., 2004).

Many types of vegetable oils can be used to produce biodiesel. Sunflower, corn, olive, palm and soybean oils are good examples of raw materials that are widely used in this process. Since Brazil is a great producer of soybean, its oil is one of the most used as cooking oil, in the food industry and in the transesterification technique (Geris et al., 2007). In addition, its composition of fatty acids is well known, consisting mostly of linoleic, oleic and linolenic acids (Kawazoe, 2014).

Several authors, such as Bernardes et al. (2007), Antunes et al. (2008) and Li et al. (2012) have worked with transesterification of soybean oil, and obtained high conversion of fatty acid esters. In these works, methanol and/or ethanol as the reaction alcohol, as well as different kinds of catalysts were used, such as potassium hidroxyde, zinc oxide, aluminium oxide and even enzymes. Dealing exactly with transesterification of soybean oil with supercritical alcohol and carbon dioxide, the papers of Bertoldi et al. (2009) and Cheng et al. (2010) can be highlighted.

In addition to the conventional method of producing biodiesel, another technique has been investigated over the past few years: the supercritical transesterification process. This method has been studied by many authors such as Demirbas (2005), Varma et al. (2010), Sawangkeaw et al. (2011), Anikeev & Yakovleva (2013), Tsai et al. (2013) and Ngamprasertsith et al. (2014). According to Marulanda (2012), this process has the advantage of promoting fast transesterification reactions of triacylglycerols and immediate esterification of free fatty acids without the excessive formation of glycerol and no need of a conventional catalyst. It also forms a homogeneous phase of the alcohol and triacylglycerols mixture.

The use of supercritical CO_2 can affect the kinetic behavior of the transesterification reaction. This may occur due to the increase of alcohol solubility in the oil phase (Galia et al., 2011). It is reported that the methylation of fixed oils using supercritical fluids may be a viable alternative for the production of biodiesel from different plant matrices (Chen et al., 2010; Soh & Zimmerman, 2011).

The type of reactor used in transesterification processes is also a very important factor to consider. Authors such as Spallina et al. (2017) and Gianotti Pret et al. (2015) worked with the design, modeling and validation of reactors. Mjalli et al. (2009) dealt with the developing and validation of a transesterification reactor. According to Kern & Shastri (2015), this process can be carried out in a continuous or batch reactor, but the batch process is often preferred because of its flexibility and accommodation of various types of raw materials, composition, and quantity, thus making possible to satisfy specific product requirements. Lim & Lee (2011) and Ghoreishi & Moein (2013) also used batch reactors to transesterificate vegetable oils using methanol in supercritical conditions, obtaining high yields of fatty acid methyl esters.

In this context, the objective of this work was to evaluate the process of supercritical transesterification of soybean oil with methanol in the presence of carbon dioxide, and provide comparisons to the conventional technique.

2 Materials and methods

2.1 Equipment description

The supercritical transesterification reaction of soybean oil with methanol and CO_2 was performed in a stainless-steel autoclave (model 2777 1000, Top Industrie, France) with internal volume of 0.095 L (0.128 x 0.152 m). To the autoclave were attached a heating collar (model 2777 1021, Top Industrie, France), a magnetic stirrer (model 616 0100, Top Industrie, FR), a cooling bath (model Q214U2, Quimis, Brazil), a thermocouple TKA 15 x 20 HMP, Erciat, France), a safety head (model 728 0350, Top Industrie, France), a rupture disk (model 728 0400, Sitec, Switzerland), a pressure transducer (model PA-Keller, Switzerland), a CO_2 supply system, a sample collection system with approximately 8 mL, and a control panel (model TS1070, Monitouch Technoshot, The Netherlands). More details are in the Appendix A at the end of the references section.

2.2 Materials

For the transesterification process, commercial refined soybean oil (LISA, Brazil), obtained in a supermarket in Belem/Para/Brazil was used as the vegetable oil. The alcohol used was Methanol (99.9% purity, TEDIA, USA). The cosolvent used was carbon dioxide (99.9% purity, White Martins, Brazil).

2.3 Transesterification method

The transesterification reaction of soybean oil with methanol and supercritical CO_2 is represented in the system shown in Figure 2. For the study, the operating conditions were those used in the paper of Han et al. (2005), who also dealt with the supercritical transesterification of soybean oil using methanol. Molar ratios of 1:24 (oil:alcohol) and 0.1 (CO_2 to methanol), rotation of 700 rpm, pressure of 130 bar, temperature of 280 °C and heating rate of approximately 4 °C/min were used. After stabilization of the selected temperature and pressure, the CO_2 inlet valve was opened so that there was no influence of temperature on the system pressure (autogenous pressure). After the reaction time (10 minutes), the sample (Figure 3) was collected and centrifuged (Multifuge X1R-N/S 41464410, ThermoElectron Led GMBH, USA) at 10,000 rpm for 15 minutes to separate the water and glycerin formed.

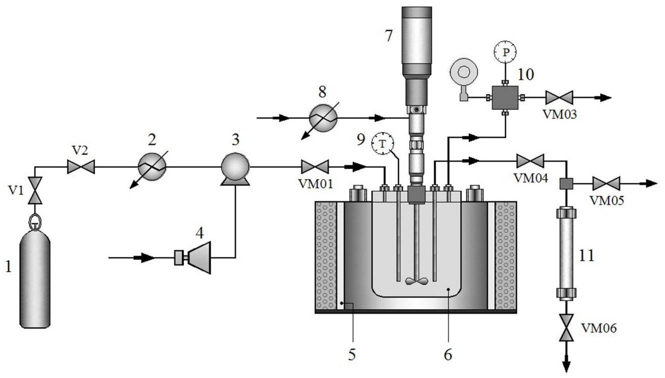

Figure 2. Schematic representation of the system used for methanolic transesterification of soybean oil. 1. CO_2 tank; 2. Cooling bath; 3. Pump; 4. Compressor; 5. Heating collar; 6. Autoclave; 7. Magnetic stirrer; 8. Cooling bath; 9. Thermocouple; 10. Safety head and rupture disc; 11. Sampler; V1-V2/VM01-VM06 Flow control valves. Described by Bezerra et al. (2018).

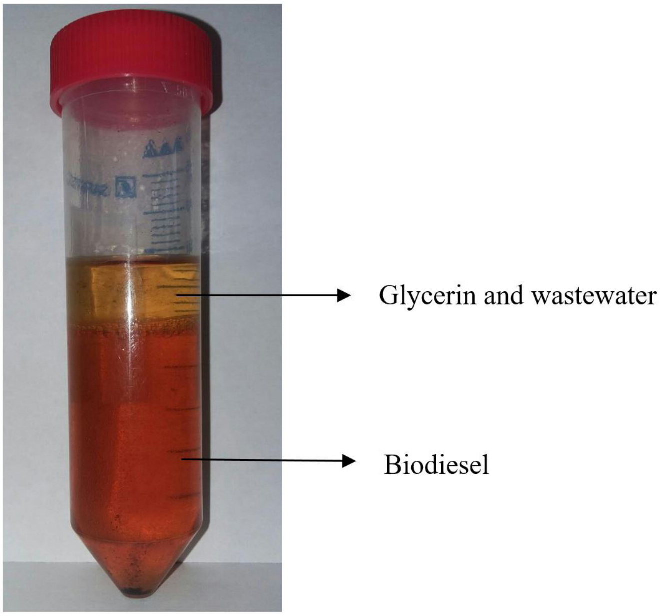

Figure 3. Sample obtained by supercritical transesterification of soybean oil with methanol and CO_2.

2.4 Analysis of the methyl esters obtained

The fatty acid methyl esters were analyzed as described in the literature (Xavier et al., 2011) by gas chromatography-mass spectrometry (GC-MS),using a QP 2010 Shimadzu system equipped with an AOC-20i auto injector, Rtx-5MS silica capillary column (30 m x 0.25 mm, film thickness of 0.25 μm) at temperatures ranging from 100 °C to 280 °C, with a gradient of 4 °C/min and injector temperature of 100 °C; helium as carrier gas at a flow rate of 1.2 mL/min (measured at 100 °C), The injector was of splitless injection, with 0.1 μL of a transesterified solution; programmed temperature of 100 °C-280 °C with a gradient of 5 °C/min. The quadrupole filter scanned from 39 to 550 Daltons once every second. The ionization was acquired by the electron impact technique with energy of 70 eV. Each chemical component was identified by comparing their mass spectra with those in the literature (Adams, 2007) and on the NIST database (Stein, 2011).

2.5 Yield of biodiesel production

The yield of biodiesel production was calculated (Equation 1) based on the fatty acid methyl esters (FAME) analyzed by GC (Baroutian et al., 2010).

$$Yield\ (\%) = \frac{A \times B}{C} \times 100 \quad (1)$$

Where: *A* is mass of biodiesel produced; *B* is total mass % of FAME; and *C* is mass of oil used in the reaction.

3 Results and discussion

3.1 Comparison to other studies on supercritical transesterification

Table 1 presents some published works on transesterification by supercritical method.

The novelty of this work is in the equipment assembly. In fact, it was specifically made with certain specifications required by the authors, for example, inlet and outlet valves for supercritical CO_2, a sampling valve, volume of the autoclave and the maximum pressure and temperature conditions that the reactor would withstand. None of the studies mentioned in Table 1 dealt with the assembly of the reactor used in their respective works. Therefore, the apparatus employed in our research is not a serial device but an exclusive equipment tailored to the authors's needs. The best conditions of pressure, temperature and molar ratio and the advantages of using methanol were considered from the study of Cao et al. (2005), which was the main paper we made comparisons with.

3.2 Influence of molar ratio

Molar ratio of alcohol to oil is one of the most significant factors that affect the conversion efficiency, yield of biodiesel and production cost (Musa, 2016). Since transesterification is an equilibrium reaction, the excess of methanol was used in order to shift the equilibrium to the right and produce more methyl esters as result of the reaction. Higher molar ratios of methanol to oil also result in a more efficient transesterification reaction, due to the increased contact area between methanol and triacylglycerols (Cao et al., 2005).

Some authors such as Van Kasteren & Nisworo (2007), Baroutian et al. (2010) and Wang et al. (2012) conducted the transesterification reactions using excess alcohol and molar ratio of methanol to oil of 24:1, which was the same proportion used in this work. At this ratio, all of them presented high yields of biodiesel produced (over 90%).

3.3 Yield of fatty acids methyl esters

In Figure 3, it's possible to see the result of the transesterification process, with a low amount of wastewater and glycerin. The biodiesel yield obtained (Equation 1) was high and equal to 95.19%, which

Table 1. Studies on the transesterification by supercritical method.

Oil	Solvent	T (°C)	P (bar)	τ (min)	MR	Y (%)	References
Soybean	Methanol	280	128	10	24:1	98.00	Cao et al. (2005)
Soybean	Methanol	320	320	75	43:1	87.50	Palacios-Nereo et al. (2016)
Castor	Methanol	300	210	90	43:1	96.50	Román-Figueroa et al. (2016)
Soybean	CO_2	85	80	60	-	92.00	Hu et al. (2015)
Rapeseed	Ethanol	350	120	20	42:1	91.90	Micic et al. (2014)
Castor	Ethanol	350	145	10	40:1	56.20	Rodríguez-Guerrero et al. (2013)
Soybean	Methanol	320	200	40	45:1	92.30	Shin et al. (2013)
Turnip	Ethanol	319	110	22	39:1	97.50	Valle et al. (2010)
Soybean	Methanol	350	150	12,5	40:1	91.40	Wang et al. (2008)
Soybean	Methanol	280	143	10	24:1	98.00	Han et al. (2005)

T: temperature; **P:** pressure; τ: time; **MR:** molar ratio; **Y:** esters yield.

was close to the results found in the literature. Han et al. (2005) obtained a biodiesel yield of 98%, using the same raw material and operating conditions of this study. Palacios-Nereo et al. (2016) also performed the supercritical transesterification of soybean oil using methanol. They reached yield of 99.9%, at molar ratio 1:42 (oil:methanol), applying gradual heating (0.51 °C/min), starting from 150 °C up to 320 °C and maintaining this temperature for 10 minutes. They found out that this hybrid process prevented the reactions of thermal decomposition while transforming all triacylglycerols into biodiesel.

According to Shin et al. (2013), in the supercritical transesterification reaction, the purification of products is much simpler, since low amount of unwanted compounds is generated, and high biodiesel yield is reached in a very short time. Thus, the technique with supercritical methanol is considered more economically viable than the use of conventional catalytic reactions.

Regarding the rate of methyl ester conversion, in the sample of oil transesterified with methanol, it is observed (Table 2) that the highest concentration is of linoleate, followed by methyl hexadecanoate, oleic acid methyl ester and methyl stearate with 41.92%, 18.07%, 7.67% and 7.2%, respectively. In general, 10 compounds were identified; together they represent 81.41% of the components present after the transesterification of the soybean oil.

In recent article, regarding production of biodiesel from vegetable matrix, Xavier et al. (2011) obtained the compounds methyl hexadecanoate (32%), methyl octadecanoate (29%) and methyl (E)-octadecanoate (29%) of *Caryocar villosum* (Aubl.) Pers, in higher concentration after the synthesis with methanol, under normal conditions of temperature and pressure. Of these components, only methyl hexadecanoate was identified in the present work in a lower concentration.

3.4 Comparison to the conventional transesterification method

Wu et al. (2013) performed the conventional transesterification process of soybean oil using methanol and CaO/NaY catalyst. They obtained biodiesel yield of 95% with 30 wt.% of CaO loaded on NaY zeolite, molar ratio 1:9 (soybean oil:methanol), temperature of 65 °C, time of 3 h and catalyst/oil mass ratio of 3%. Liu et al. (2008) also dealt with biodiesel production from soybean oil and methanol, using CaO as a solid base catalyst. Assays at molar ratio 12:1 (methanol to oil), addition of 8% CaO, and 65 °C gave the best results (over 95%), in 3 h.

Table 2. Fatty acid methyl esters identified in the transesterified product. Composition obtained by gas chromatography coupled to mass spectrometry.

RT(min)*	RI**	Fatty acid methyl esters	wt (%)
5.325	1114	Methyl octanoate	1.22
11.000	1323	Decadienal<2E,4E->	0.62
26.258	1725	Tetradecanoate<methyl->	1.12
34.017	1945	Methyl hexadecanoate	19.07
35.467	1989	Hexadecanoicacid<n-	0.02
39.100	2102	Linoleate <methyl->	41.92
40.325	2141	Oleic acid methyl ester	7.67
40.675	2153	Methyl stearate	7.2
45.183	2303	11-Eicosenoic acid, methyl ester	1.27
51.483	2531	Docosanoate<methyl->	1.3
Total			81.41

*RT: Retention time; **RI: Retention Index.

In both studies, the biodiesel yield obtained was close to that reached in this work. Despite the fact that lower reaction temperatures and molar ratios were necessary, with no need of increasing pressure, they had to use base catalysts and reaction time was quite superior to that applied in this study. Also, fewer residues are generated, and the product purification is performed in fewer steps. However, it cannot be said that the supercritical transesterification is economically viable compared to the conventional technique, due to energy and equipment costs are higher than those observed in a conventional process. Thus, a comparative economic analysis is necessary.

3.5 Influence of the reactor type used in the transesterification process

Silva & Oliveira (2014) state that the biodiesel production under supercritical conditions requires high processing costs (high molar ratios, pressures and temperatures) and some strategies to reduce the expected high operating costs usually involve the addition of cosolvents and the use of different reactor configurations with increased mass transfer, operating in continuous mode (microreactor or packed bed reactor). Many authors such as Pohar & Plazl (2008), Hessel et al. (2005), Wen et al. (2009) recommend to conduct transesterification reactions in microreactors in order to reduce the limitations of mass and heat transfer, since such parameters tend to increase due to the small size and large contact area (Pohar & Plazl, 2008), and the small internal diameters (typically 10-300 μm) promote interaction with the reagents at molecular level. In the case of the reactor used in this study, its internal diameter (51 mm) and volume (0.095 L) represent the most suitable configuration, which was verified by the high biodiesel yield obtained (95.19%).

Comparing to other studies, Wang et al. (2008), for example, used a continuous tubular reactor with no mechanical blending to perform the transesterification reaction of soybean oil with methanol under supercritical conditions. Thus, in order to increase the biodiesel yield, they had to acidify the soybean oil by adding a certain amount of organic into the reaction system. At the molar ratio of 1:20 (oil:methanol), the biodiesel yield of acidified oil, and non-acidified oil were 82.3 and 60.0%, respectively.

4 Conclusion

The reactor used in this study proved to be efficient for the transesterification process. The reaction time was only 10 minutes, under experimental conditions of 130 bar, 280 °C and molar ratio of raw material to methanol of 1:24, and CO_2 to methanol of 0.1. The yield of biodiesel was 95.19% and the yield of methyl esters was 81.41%, with linoleate being the compound found in the highest concentration. The results indicate that the undesirable compounds glycerin and wastewater were obtained in low concentrations, which means that this method can be applied to obtain biodiesel with no need of base catalysts and fewer steps of purification compared to the conventional method.

Acknowledgements

Wanessa Almeida da Costa (1427204/2014), Fernanda Wariss Figueiredo Bezerra (1718822/2017), Mozaniel Santana de Oliveira (1662230/2016), Marcilene Paiva da Silva (1636612/2016), and Vânia Maria Borges Cunha (1566277/2015) thank CAPES for the scholarship. The authors thank FAPESPA (ICCAF: 134/2014).

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Apenddix A. Equipment description

1 Autoclave characteristics

1.1 Technical datasheet and marking

Table A1 shows specifications such as brand, model, size and operating range of pressure and temperature.

Table A1. Autoclave characteristics.

Manufacturer	Top Industrie	
Description	Autoclave	
Type	Autoclave (2777 1000) Off Category according to PED 97/23 (Pressure Equipment Directive, 1997) ($v < 0.1$ L) Serial Number - 2986	
Year of Manufacture	2015	
Production Site	FR – 77 013 VAUX LE PENIL	
Selected design method	CODAP 2005 Division 2 + Rev. 09/07 and 03/09	
Evaluation procedure	A Module	
Dimensions	∅ 128 x 152 mm	
Internal Volume (V)	0.095 L	
Operating Pressure (PU)	300 bar	
Maximum Allowable Pressure (PS)	400 bar	
Pressure Test (PT)	667 bar	
Operating Temperature (TU)	10 °C to 350 °C	
Design Temperature (TS)	10 °C to 400 °C	
Material		**Drawing**
	Body: 1.4571 according to NF EN 10272 (Le Bureau de Normalisation, 2000)	Normes 2777 1020
	Plate: 1.4571 according to NF EN 10272 (Le Bureau de Normalisation, 2000)	Normes 2777 1013
	Ring: 1.4980 according to NF EN 10269 (Le Bureau de Normalisation, 2013)	Normes 2777 1012
	Nut: 1.4980 according to NF EN 10269 (Le Bureau de Normalisation, 2013)	Normes 2777 1010)
Type of fluid	Group 1	

1.2 General characteristics

Figure A1 shows the autoclave in its structural frame coupled to the gas supply system and stirrer with water for its cooling system. Table A2 shows the dimensions of the structure with the autoclave.

Figure A1. Frame with autoclave.

Table A2. Measures of the frame with autoclave.

Magnitude	Approximatesize
Width(mm)*	51
Height(mm)	85
Depth (mm)	42

*(not including the 4 ground fixing squares).

2 Design

2.1 Control cabinet

From the control cabinet (Figure A2), it's possible to do: **i.** reading and regulation of the stirring speed; **ii.** temperature reading and regulation; **iii.** pressure reading; iv. flow measurement; v. emergency stop button.

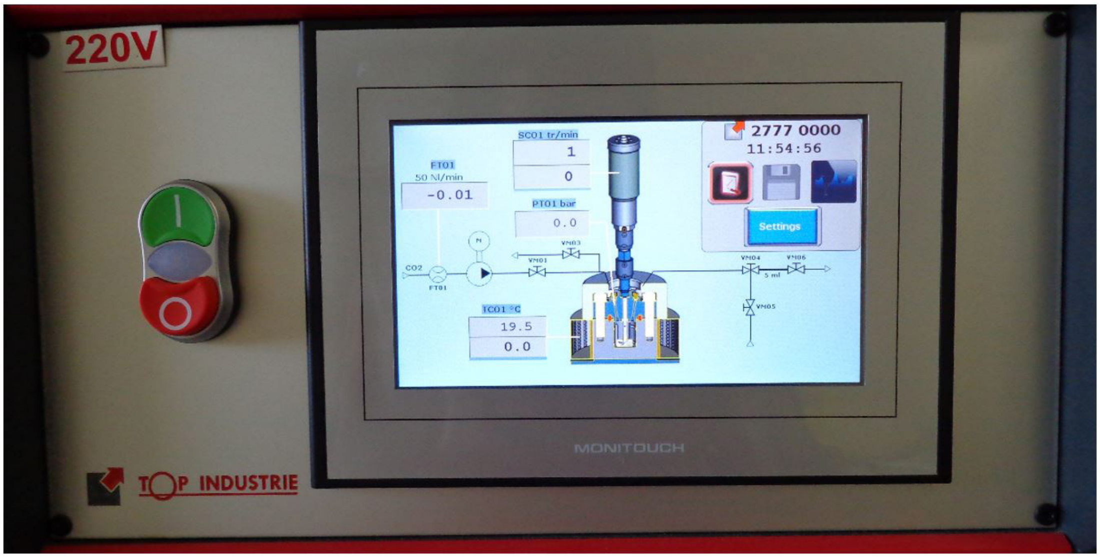


Figure A2. Control cabinet.

The control cabinet has the following features:

- Electrical supply: 230 VAC / 50-60 Hz / 16 A
- Maximum power: 3000 W

3 Assembly of the autoclave

For correct assembly of the autoclave it is necessary to perform the following procedures:

- Check the property of the surfaces receiving the seal;
- Pull of the seal and clean it;
- Check the property of the threads on the body and the ring;
- Put if possible high temperature grease on the threads;
- The liquid should never completely fill the autoclave;
- Close the autoclave and tighten with the torque wrench showed in Figure A3 (Facom/nS.306A100 + extension S.210 + socket Hex 24mm S.24H), in cross with tightening torque 20 N.m, then tightening torque 50 N.m (see Figure A4 for the detail of tightening in cross);
- Make if possible a tightness test at room temperature;
- Mount the insulating mantle on the top of the autoclave.

Figure A3. Torque wrench to close the autoclave.

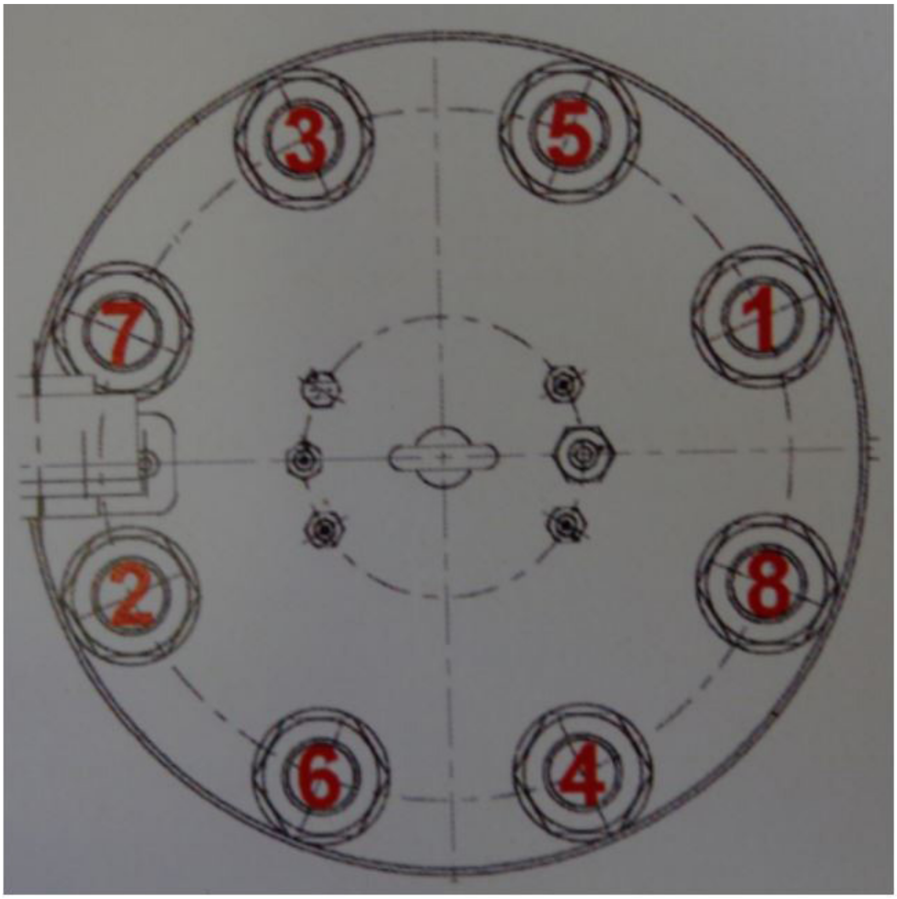


Figure A4. Detail of tightening in cross to correctly close the autoclave.

Observation: Never scratch contact surfaces from body or plate with a metal object, above all the effective cover area of gaskets. Never loosen or tighten up connections under pressure.

4 Operating of the autoclave and control cabinet - Standard Operating Procedures (SOP'S)

4.1 Detailsofthe autoclave

The Figure A5 shows the location of the pressure transmitter, the safety head and disc, the manual valves, the mechanical boy and the floor fixing squares.

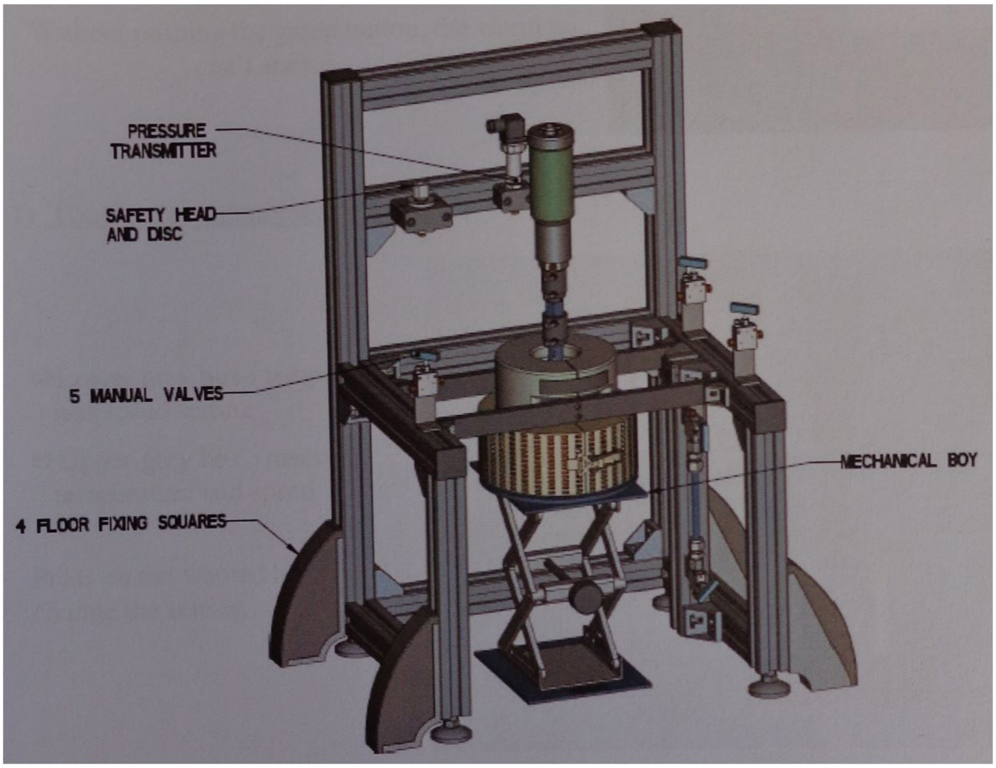


Figure A5. Details of the autoclave.

4.2 Use of the control cabinet

To use the control cabinet is required:i. powering of box by the master switch on the rear side (Figure A6.a); ii. start of the general by the green button “I” (Figure A6.b) without pushing the green button, the warm up can´t start.

Figure A6. Control cabinet bottom (a) and front part (b).

Touchscreen general screen

The screen presents two main boxes for controlling of temperature and speed (Figure A7):

- Upper-grey box: measured temperature and speed. It’s necessary to press on the wanted box no change the settings (“A.1”);
- Lower grey box: temperature and speed setting (“A.2”).

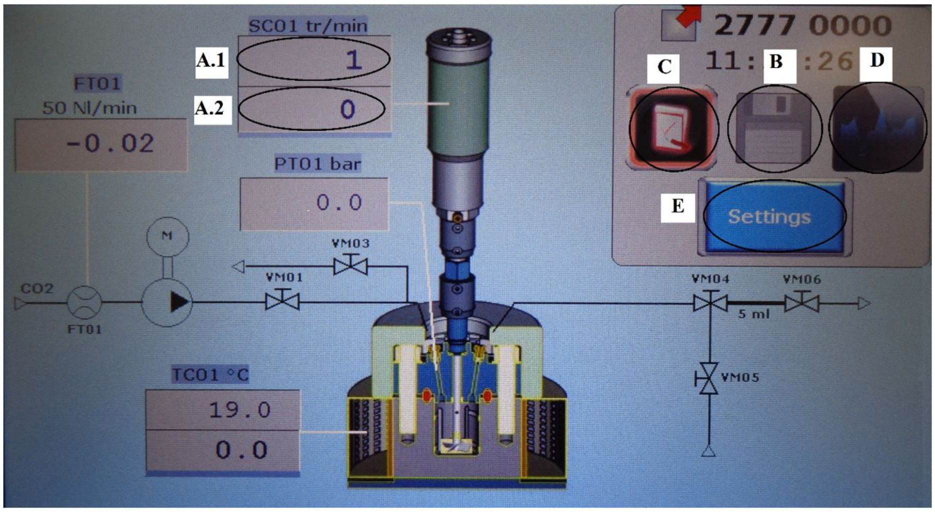


Figure A7. Touchscreen cabinet control.

Notes:

- To save the data, insert the USB key before the beginning of test to begin the recording of the test data (USB connection beside the box) – No data storage without USB key.
- Press on "B" (Figure A8) to start the recording of the temperature value;
- Press on "C" (Figure A8) to go to the "Warring screen";
- Press on "D" (Figure A8) to go to the "Curves screen".

Setting screen

Press on the "Settings" (Figure A8 - "E") on the general screen:

- Press on the wanted box to have access to the HOUR screen (Figure A8 – "A");
- Modify the values with the boxes "+" and "-", then validate on "B" and go back to the general screen by pressing on "C" (Figure A8).

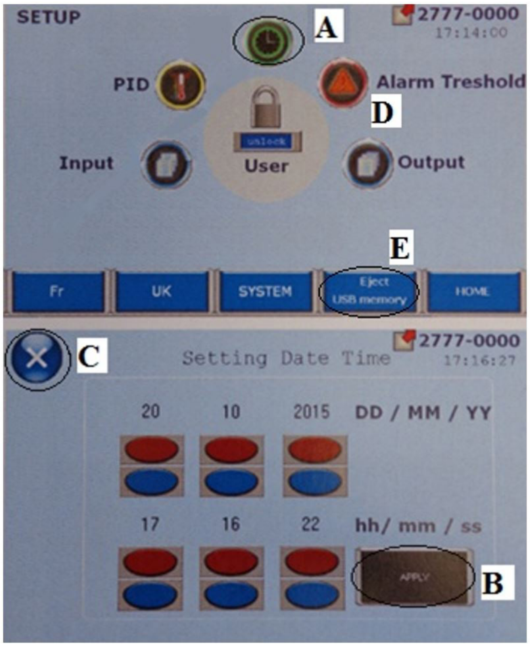


Figure A8. Modifying some settings.

Curves screen

To go to curved screen is necessary to press the “D” icon (Figure A8).

- In the graphic boxes press on the icons “A” for 30 seconds, return to box and press on “B” for 5 seconds and return (Figure A9);
- Press the icon “Graph return” – “C” (Figure A9) to return at real-time;
- To change the scale of the Min or Max values: i. press on the value to change; ii. enter the new value on the keyboard; iii.validate by “OK”.

	SV TC01	TE01 °C	TE101 °C	SV SC01	SC01 rpm	I mot	PT01 bar	FT ml/min
max	400.0	255.0	500	1500	1500	2.00	320.0	60.00
min	0.0	0.0	0	0	0	0.00	0.0	0.00
val	0.0	22.0	22	0	1	0.00	0.0	-0.02

Figure A9. Curves screen.

Ejection of the USB key at the end of the test to get back the test curves

- Press on “Eject USB Memory” (Figure A8 – “E”) and wait for the flickering of the paving stone.
- Storage of the data in the file DA T0000\SAMPLES\SMP0000.csv

Note: the ejection of the USB key stops the posting of the curves on the screen

Warning screen

Press on “C” (Figure A8) to reach the screen. In this screen (Figure A10) you see information about alarm, alarm list, hours of start hour of ending. If the hour of ending is not written, the alarm is still continuing.

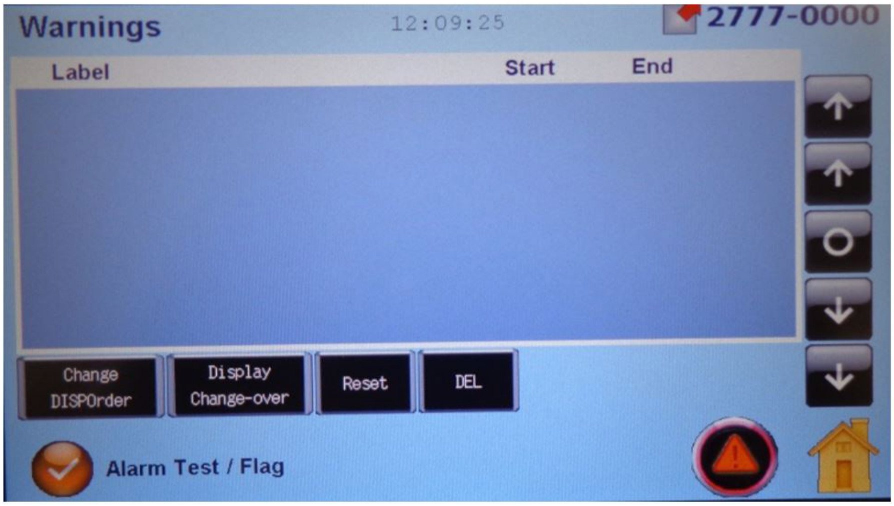
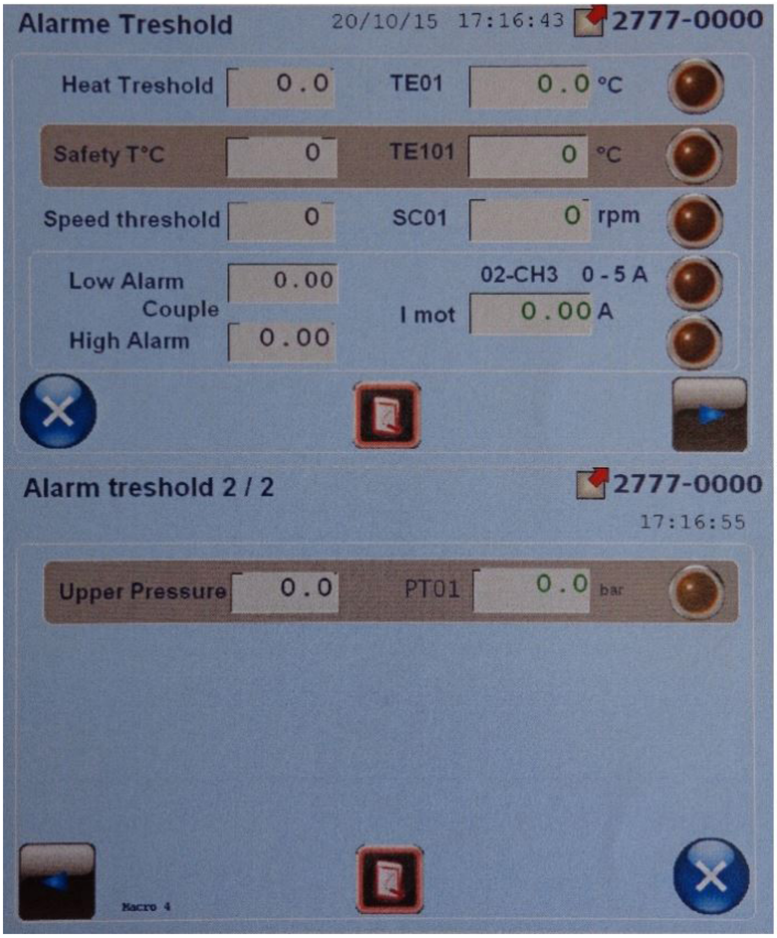


Figure A10. Warning screen.

Alarm threshold

To access this function press on the settings screen (Figure A8 - "D") to reach this screen (Figure A11).

In this screen you can put alarm on different temperatures (right box in green) and measured temperatures (left box in blue).



Alarme Treshold 20/10/15 17:16:43 2777-0000

Heat Treshold 0.0 TE01 0.0 °C

Safety T°C 0 TE101 0 °C

Speed threshold 0 SC01 0 rpm

Low Alarm 0.00 02-CH3 0 - 5 A

Couple

High Alarm 0.00 I mot 0.00 A

Alarm treshold 2 / 2 2777-0000 17:16:55

Upper Pressure 0.0 PT01 0.0 bar

Figure A11. Alarm Threshold.

5 Maintenance

5.1 Principles for high pressure parts

- Pipe connections:
  - Make sure that the connection is equivalent to the desired pressure;
  - Increase pressure by stages and check the mechanical resistance;
  - Never look for a leak using the hands;
  - Never stand in front of a connection which has just been made;
  - All connections shall be tested at maximum operating pressure before normal operation.
- Put a plug on all openings not used (circuit cleanliness).
- All used connections shall be referenced showing the operating pressure.
- List the internal connections;
- All connecting capillary tubes shall be fitted with a whiplash prevention system.

5.2 Autoclave 2777 1000

Before carrying out maintenance, each time check that appliance is at ambient temperature and zero pressure using the pressure and temperature sensors located on the appliance.

Annual preventive maintenance

- Check the inside and outside parts under pressure to look for any damage or signs of corrosion.
- Check the safety organ (we recommend to change the safety disc).
- Check the pressure transmitter and the temperature measure loop.

Corrective maintenance

- Check the gasket faces in the autoclave in the event of a leak, and stripe impose a machining repairing.

5.3 Maintenance expected for spare parts:

The designation, size, supplier and use of spare parts are shown in the Table A3.

Table A3. Spare parts.

Unit	Designation	P/N	Supplier	Use
2	O-ring viton	∅ 10.82 x 1.78	Sephat or equiv.	Stirrer internal
2	O-ring viton	∅ 14 x 1.78	Sephat or equiv.	Stirrer internal
3	Bearing	604 01 40 1192N	TOP INDUSTRIE	Stirrer internal
2	O-ring	2659 2005	TOP INDUSTRIE	Stirrer/autoclave
2	Plate seal	R 18 OCTOGONAL -POUR BRIDE RT	DELTA FLUIDE	Autoclave seal
4	O'ring Viton	∅ 20.35 X 1.78	Sephat or equiv.	Water jacket seal

5.4 Stirrer series 618 01 00

Bearings

- If the stirrer becomes noisy and/or vibrates, the bearings must be changed;
- If the noise and/or the vibrations persist, the stirrer must be returned to our after-sales service;
- The transversal groove on the bearing must not be in contact with the encapsulation.

Monthly preventive tightness maintenance

- O-ring seal marker 8: if the 'O' ring seal marker 8 is faulty, the gas leak will be canalized into the leak hole of the distance sleeve marker 2 (Figure A12). Use a bubbling product (water and soap) to control whether the O-ring is perfectly thight/fitting;
- Seal (between part marker 2 and flat bottom): use a bubbling product (water and soap) directly on the connection (M20 x 150) between the stirrer and the autoclave head (Figure A12);
- Motor, ball bearing, magnetic coupling: no maintenance. If a problem appears, the complete stirrer must be returned to our after-sales service.

Annual preventive maintenance

- Change the bearing and the O-ring;
- Check the magnetic coupling efficiency;
- For maximum protection, the stirrers are designed so that the compressed air or electric motor blocks and not the magnetic coupling.

Corrective maintenance

- If the stirrer becomes too noisy or vibrate, change the bearings;
- If the noise still exists, the stirrer must be returned to the SAV service for repairing.

Corrosion: The parts that are in contact with the fluid are made of stainless steel 1.4404 and 1.4980. No traces of corrosion should appear. If this were to happen, it would be because the equipment was not suitable for application. It is dangerous and forbidden to use a stirrer under such conditions.

Dismounting

- Disconnect the stirrer from its power or energy supply;
- Disconnect the stirrer from the autoclave;
- Unscrew and remove the screw marker 6 (Figure A12);
- Unscrew the motor-distance sleeve (marker 12) while holding the body (marker 2) and the ring marker 9; apply pressure slightly in order to defeat the attraction of the magnetic field (Figure A12);
- Unscrew the ring number 9 while holding the body marker 12 (Figure A12);
- Pull the upper body marker 11 towards the top. Remove the stirring module;
- The central axis marker 9 (Figure A12) may now be removed from its housing;
- The bearings (marker 1) and the O-ring (marker 8) are now accessible for checking or replacement (Figure A12).

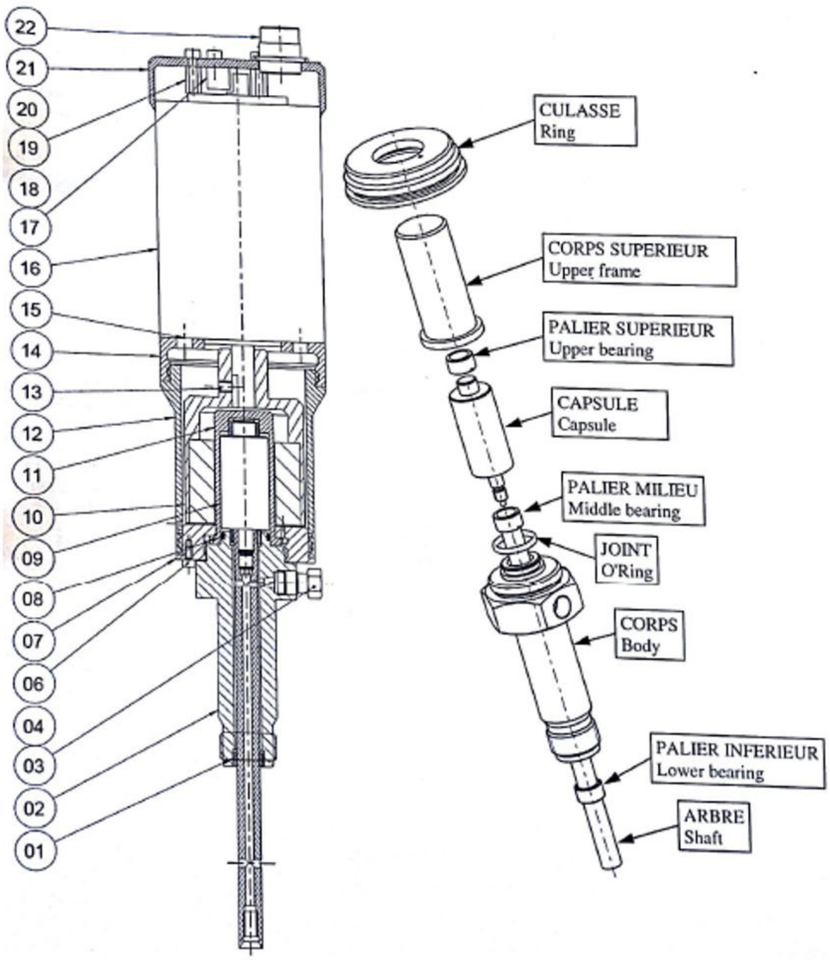


Figure A12. Stirrer's parts.

Assembly

After having thoroughly cleaned all the parts and having, if necessary, proceeded to replace the bearings marker 1 and the seal marker 8 (Figure A12), reassemble as follows:

- Slide the central axis marker 9 (Figure A12) into its housing and manually, make sure that the it rotates correctly;
- Put marker 11 (Figure A12) back into place by forcing slightly. If permitted, it is advisable to apply a drop of lubricant such as vaseline in order to make sliding easier on the joint;
- Manually screw back on the part marker 12 (Figure A12). Tighten until metal against metal contact is obtained;
- Rescrew the screw marker 6 (Figure A12).

Note: for the P/N of the bearings and O’ring, see the autoclave part list.

5.5. Settings of parameters on the touchscreen:

Accessible by pressing on the button *Settings* (Figure A8 - “E”). The settings are locked by default (“lock”). To unlock, press on the ‘unlock’ box (Figure A13 – “A”); when the next screen appears, enter the password (TOP) and validate by “Enter” and press on “B” (Figure A13) to go back to the *Setting screen*.

Note: Any parameter modification must be made only by qualified person.

Figure A13. Unlocking the settings.

Technical data

Electrical characteristics

Power supply voltage Vcc (tolerated ripple < 5% ………………………..10 to 60 DVC

Permanent output current I_{mom}……………………………………………………10 A

The motor current is limited to I_{mom} + I_{boost} for 500ms then to $I_{mom}$for 10s (motor overload) before being cut (motor locks up). Any current return under the limitation resets the detection time

Commutation frequency………………………………………………………. 20 KHz

Inputs

SET VALUE: Set point value (terminals 1, 2 and 3)………...**0/+10 DVC** (R_t> 10 KΩ)

ENABLE: active when the contact is closed (terminals 4 and 8)……......... **Dry contact**

CW/CCW: open contact: clockwise (terminals 5 and 8) contact closed: Counterclockwise direction………………………………………………. **Dry contact**

In the event of an inversion of the rotation direction with a non-zero speed, the servo amplifier waits until the current is cancelled in the motor before reversing the rotation direction.

Outputs

I MOT: Image with I_{mom} current voltage during nominal operation (terminals 9 & 12)

This output is protected against short-circuits………………...**0/ + 10 VDC** (R_0 = 100Ω)

Proportionality factor……………………………………………………………**1V/2A**

MONITOR: Image with I_{mom} and I_{boost}current voltages during settings (terminals 10 & 12)

This output is protected against short-circuits…………………**0/ + 5 VDC** (R_0 = 100Ω)

Proportionality factor……………………………………………………………**1V/2A**

READY: Message for monitoring the state of card (terminals 11 and 12)

Open collector not protected from the short-circuits………..**max. 30 VDC** (I_t < 20 mA)

Output voltage

+ 10V: Auxiliary voltage (terminals 1 & 3) ……………………**+10 VDC,** max. 20 mA

Protected against short-circuits and overloads

Setting Push buttons

OK: Validation or setting mode input/output

+: Increase of parameterisable values

+: Reduction of parameterisable values

LED indicador

Green LED…………………………………………………………...Presence of voltage

Orange LED Steady……………………………………...Clockwise rotation direction

Orange flashing LED………………………………Counterclockwise rotation direction

Red LED……………………………………………………………………….Fault

Temperature / Humidity

Operation…………………………………………………………………..**0 to +45 °C**

Storage……………………………………………………………………..**-40 to + 85 °C**

Relative humidity…………………………………………**20 to 80% non-condensed**

Mechanical characteristics

Weight………………………………………………………..**approximately 220 g**

Dimensions………………………………………………..**102 x 100 x 32 (or 3U frame)**

Mounting plate…………………………………………………………..**for M4 screw**

Connections

Terminals with screws**………………………………… "Power"(4 poles), "Signal"(12 poles)**

Power step…………………………………………………………………….**5.08 mm**

Suitable for sections of wire……………………………………………**0.5 to 2.5 mm^2**

Signal step……………………………………………………………………**3.81 mm**

Suitable for sections of wire…………………………………………..**0.14 to 1.5 mm^2**

The Table A4 shows the parts list as well as their material and suppliers.

Table A4. Parts list.

	Nb	*Designation*	*Material*	*Ref. Top*	*Ind.*	*Ref. Supplier*	*Supplier*
01	1	Magnetic stitrrer 30 N.cm		616 01 00	D		Top Industrie
02	2	Water Jacket		604 99 00	C		Top Industrie
03	1	stirrer adapter	1.4404	269 1004			Top Industrie
04	1	Stirrer seal	Viton			∅10.82 x 1.78	Sephat
05	4	1/8" HP Screw		920 01 00			Top Industrie
06	4	1/8" HP Ferrule		920 02 00			Top Industrie
07	1	Mantile insulator		2777 1007	A		Top Industrie
08	1	Stirrer seal washer	1.4404	2777 1008			Top Industrie
09	1	Stirrer seal	NIKEL	2659 2005	A		Top Industrie
10	8	Nut - M16 x 1.5	1.4980	2777 1010			Top Industrie
11	8	Washer M16	INCO 718	692 02 16 INCO 718			Top Industrie
12	8	Stud – M16	1.4980	2777 1012			Top Industrie
13	1	Flat bottom	1.4571	2777 1013			Top Industrie
14	1	Flat seal	Stainless stell			R 18 Octogonal RTJ flange	Top Industrie
15	2	Lower bearing	1192N	604 01 40	B		Top Industrie
16	1	Hc M3 X 8 mm Screw				Hc M3 X 8	Top Industrie
17	1	DB baffle bar	1.4404	653 00 DB 00			Top Industrie
18	1	Stirring shaft	1.4404	2777 10 18			Top Industrie
19	1	∅ 20 Impeller		608 01 94 00			Top Industrie
	1	Body impeller	1.4404	608 01 94 01			Top Industrie
	8	bar	1.4404	608 01 94 02			Top Industrie
20	1	Body	1.4571	2777 10 20			Top Industrie
21	1	Heating collar		2777 10 21			Top Industrie
22	2	FHc M8 x 25 mm (high temperature) Screw	Stainless stell			Fhc MB x 25	BAFA
23	1	Botton insulator	Mica silicone	2777 10 23	A		Top Industrie
24	1	Thermocouple K ∅ 1.5 – L 200 mm				TKA 15 x 20 HMP	Erciat
25	1	Thermowell	1.4404	2777 10 23			Top Industrie

Table A4. Continued...

	Nb	*Designation*	*Material*	*Ref. Top*	*Ind.*	*Ref. Supplier*	*Supplier*
26				**Pieces de rechance**			
27	2	Stirrer seal	Viton			∅10.82 x 1.78	Sephat or equiv.
28	2	Stirrer seal	Niquel	2659 2005	A		Top Industrie
29	9	Botton flat seal	Inox			R 18 Octogonal RTJ flange	Delta Fluide
30	3	Lower bearing	1192N	604 01 40	8		Top Industrie
31	1	Torque key (50 N.m)				S.308 A 100	Facom
32	1	Ratchet				S.210	Facom
33	1	Hexagonal socket M 16 / HEX 24 mm				S.24H	Facom
34	3	Lower bearing	Graphite	604 01 40 GR	B		Top Industrie
35		O'ring (internal stirrer)	Viton			∅14 x 1.78	Sephat or equiv.
36		O'ring (water jacket)	Viton			∅ 20.35 x 1.78	Sephat or equiv.