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SUPERCRITICAL CO_2 EXTRACTION PILOT PLANT DESIGN – TOWARDS IoT INTEGRATION

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Preliminary communication

The interest in high pressure technology during last decades increased intensively. Supercritical Fluid Extraction (SFE) is a process that is growing in importance as an alternative to conventional separation processes. SFE uses environmentally friendly CO_2 as the extracting agent in the process because of its relatively low critical pressure (7,38 MPa), its low critical temperature (304 K), its non-dangerous character and low cost. During this process it is necessary to use high pressures in the procedure. The extractor vessel (pressure vessel) is the most important equipment of the system, where the supercritical conditions need to be established and the extraction occurs. Also other devices (separator vessel, heat exchangers, valves etc.) are necessary to be involved in the process due to used high pressures. Safety is the most important factor while dealing with SFE systems and the design of such equipment with full safety of process is very hard task. Therefore, to achieve the high desired safety level, a reliable control system must be designed as the control system and data communication segment. Various different process parameters such as CO_2 mass flow rate, extraction pressures and temperatures affect the extraction process and the quality of the extract; hence these parameters need to be precisely controlled and monitored during the extraction. A design of one supercritical CO_2 extraction laboratory-pilot plant and development of a remote control and its supervision system is presented in this paper. The developed SFE system (mechanical and electrical components) was compared with the existing commercial systems and its main advantages over the existing systems are presented. By enabling remote control and supervision the classical process control is joined with the concept of Internet of Things (IoT), where the information becomes omnipresent in the vast realm of Internet.

Keywords: *embedded system; process control; supercritical fluid extraction; system construction.*

Projektiranje pilot postrojenja za ekstrakciju superkritičnim CO_2 – prema integraciji s Internetom objekata (IoT)

Prethodno priopćenje

Interes za tehnologije visokog tlaka tijekom posljednjih desetljeća se intenzivno povećava. Ekstrakcija super kritičnim fluidima (SFE) je process koji predstavlja alternative konvencionalnim postupcima separacije. U procesu ekstrakcije superkritičnim fluidima koristi se ekološki CO_2 kao ekstrakcijsko otapalo zbog relativno niskog kritičnog tlaka (7,38 MPa), niske kritične temperature (304 K), poželjnih svojstava i niske cijene. Tijekom ovog postupka, potrebno je rabiti visoke tlakove. Ekstrakcijska posuda (posuda pod tlakom) je najvažnija oprema sustava, gdje se trebaju ostvariti kritični uvjeti i gdje se odvija ekstrakcija. Također, u procesu se treba obratiti pažnja i na druge dijelove uređaja (separator, izmjenjivače topline, ventile i sl.) zbog uporabe visokih tlakova. Sigurnost je najvažniji factor kada se radi o SFE sustavima i projektiranje takve opreme s potpunom sigurnosti procesa predstavlja vrlo težak zadatak. Stoga, kako bi se postigla visoka razina sigurnosti, pouzdan sustav kontrole mora biti osmišljen kao komunikacijski segment sustava kontrole i podataka. Različiti procesni parametri, kao što su protok CO_2, tlak i temperature ekstrakcije, utječu na process ekstrakcije i kvalitetu dobivenog ekstrakta. Stoga ovi parametri trebaju biti precizno kontrolirani i nadzirani tijekom ekstrakcije. Projektiranje jednog laboratorijskog-pilot postrojenja za ekstrakciju superkritičnim CO_2, te razvoj daljinskog upravljanja i nadzora sustava prikazani su u ovom radu. Razvijeni sustavi SFE (mehaničke i električne komponente) uspoređeni su s postojećim komercijalnim sustavima, gdje su prezentirane njegove glavne prednosti u odnosu na postojeće sustave. Omogućavanjem daljinskog upravljanja i nadzora klasična kontrola procesa je spojena s konceptom Interneta objekata - Internet of Things (IoT), gdje informacija postaje sve prisutna u ogromnom području Interneta.

Ključne riječi: *ekstrakcija superkritičnim fluidima; kontrola procesa; projektiranje; ugradbeni računalni sustav*

1 Introduction

The objective of every food production is to achieve high quality, minimally processed, "natural", additive-free food with high nutritional value. Researchers have been trying to find the best alternative processes to minimize the environmental impact, decrease the toxic residues, more efficiently use sub-products and also produce higher quality foods. The oil extraction with supercritical fluids is an alternative method to replace or to complement the conventional industrial processes such as pressing and solvent extraction. The traditional organic solvent extraction methods are very time-consuming; they require relatively large quantities of solvents, leave toxic solvent residue, and cause degradation of unsaturated compounds due to the heat. Due to this fact, there is an increasing demand for different extraction techniques with shorter extraction time, reduced organic solvent consumption, and decreasing pollution. Supercritical Fluid Extraction (SFE) technique presents various advantages over traditional methods, such as the use of low temperatures, reduced energy consumption and high product quality due to the absence of solvent in extracts. Carbon dioxide (CO_2) is the most widely used compressed fluid because it is non-toxic, non-explosive, inflammable, cheap, readily available, easy removable from the product and possesses moderate critical properties (T_c = 31,1 °C, P_c = 7,38 MPa) [1]. The technical and environmental advantages of the SFE technology, as well as the fact that CO_2 is generally recognized as a safe (GRAS) solvent, should be utilized in as many industrial applications including also the vegetable oil extraction. The disadvantages of the SFE technology are the equipment acquisition high investment costs and the high energy demand of the CO_2 extraction unit [2÷7].

Handmade supercritical fluid extraction (HM-SFE) system enables the extraction in an inexpensive way. The obtained extraction yields and composition are very similar to those obtained by the commercial SFE system [8]. Just like the commercial supercritical fluid extraction systems, the handmade supercritical fluid extraction system is composed of various components that need to be tuned to achieve the optimal extraction process. The first step of the design approach for the SFE system design is to define requirements for an electronic system (pressures, temperatures, etc.). Secondly, the data acquired by the electronic system are presented to the end user by means of the designed accompanying applications. Parameters such as temperatures, pressures and flow rates of fluid need to be monitored and controlled in order to enable the efficient and economical extraction process. Due to the fact that the aforementioned parameters affect the extraction yield and the quality of the extract [9], these parameters need to be controlled and monitored with high precision. Since the process of SFE has been used on industrial scales for decades, the constraints on regulation and control are now well known. However, with the arrival of new communication technologies and the concept of Internet of Things (IoT) [10], the ability of remote multi-parametric monitoring and control of the SFE process is again brought into focus. It is expected that the number of devices connected to the Internet in future will grow exponentially. To be precise, in 2011 the number of interconnected devices on the planet overtook the actual number of people. Today more than 9 billion devices are present on the planet, and by some forecasts in year 2020 will be more than 24 billion devices present on the planet [18]. Big number of the present devices interconnected among each other within Internet results in abundant exchange of information between the sole devices (Machine to Machine communication, M2M) and between humans and devices.

As the application of the remote control and monitoring system is targeted at low cost HM-SFE systems used for research, the design of the electronic system must be adapted to these constraints. Furthermore, the system must contain safety features with the ability of emergency system shutdown due to system failures. Finally, the data from the SFE system were disseminated to the end user, where the comparison with the commercial systems was performed. The comparison of the system was achieved by measuring extraction yield during a period of time. The results obtained with the HM-SFE system were compared with the commercial SFE system (HPEP, NOVA-Swiss, Effertikon, Switzerland) in the extraction of oil from soybeans.

The paper is structured as follows. Section 2 deals with the mechanical design of the SFE system. The construction of new HM-SFE system is shown in this section and each part of equipment is explained in details. Section 3 provides the detailed description of the designed supporting electronic system alongside with the projection measurements. Also, the communication aspect, graphical interface and the developed application are presented. Finally, Section 4 provides the comparison of the HM-SFE system performed with the existing commercial system, followed by the conclusions in Section 5.

2 Handmade supercritical fluid extraction (HM-SFE) system

The basic functionality of SFE is the ability to use CO_2 gas as a solvent, passing through a material and extracting the desired components. In this process the gas is pressured at high pressures and heated to achieve supercritical state. After the gas is supercritical, it passes through the material (in the extraction vessel) extracting the desired components. Finally, the components are separated from the gas in the separator leaving the extract in a cuvette and the gas is released into the atmosphere (or recuperated). The process diagram of the SFE and the constructed system used for supercritical fluid extraction is given in Fig. 1. The construction and assembling of the HM-SFE system was performed by Đuro Đaković Aparati Inc. (Slavonski Brod, Croatia) which conducted the material durability tests and a pressure test for vessels. The materials used for the construction of the HM-SFE system are stainless steel AISI 316Ti and AISI 304. All additional connection tubing parts are also the same grade of material. Extraction and separator vessels were properly tested at safety factor of 1,5. The extraction vessel was tested at working pressure 50 MPa and the separator vessel at 3 MPa.

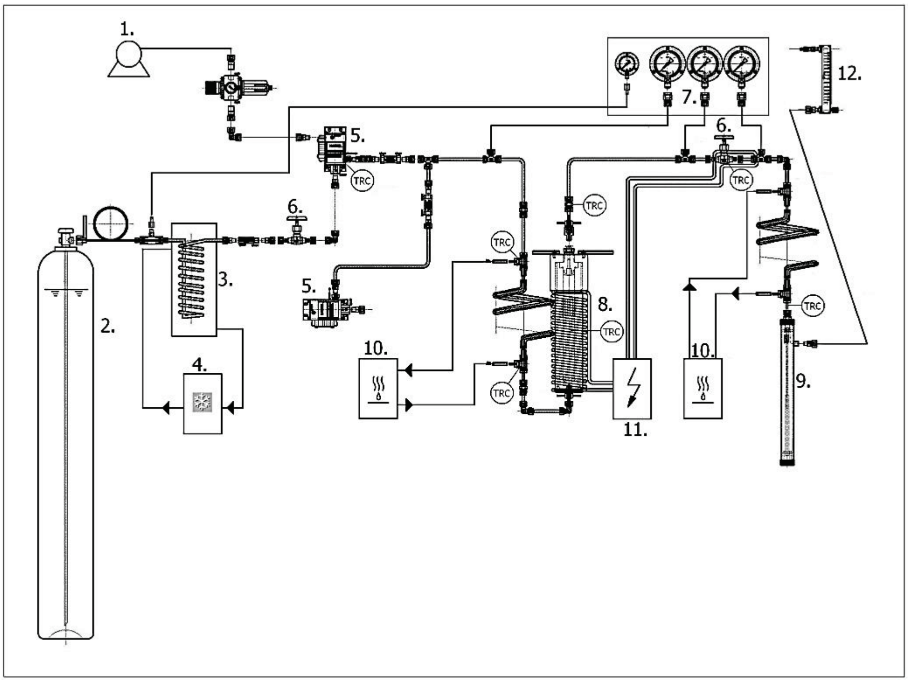

Figure 1 Handmade supercritical fluid extraction system
(1. Compressor; 2. CO_2 tank; 3. Stainless steel coil; 4. Cooling bath; 5. Air driven fluid pump Haskel MS-71; 6. Valves (B-HV); 7. Manometers; 8. Extraction vessel; 9. Separator vessel; 10. Water bath; 11. Centralized system glass fiber heater; 12. Flow meter)

The extraction vessel was made of a stainless steel bar (AISI 304) O.D. 100 mm and height 500 mm. A stainless steel rod is drilled (center hole) with a 40 mm bore for a 400 mm. The upper inside part of an extraction cell was polished to plug well gaskets. The cap of an extraction cell was designed to hold plug and it is connected with the extraction cell through a trapezoidal thread. The plug was patented by the company that assembled the HM-SFE system and sealed in two places with an O-ring. The lower part of the extraction cell was also drilled and prepared for quick connection with R connector and the O-ring seal. The separator vessel is made of a stainless steel seamless tube (AISI 304) 50×5 mm. It has two plugs at upper and lower side of the separator. The plugs are sealed with the O-rings to ensure gas tightness. The lower plug is made as a holder of a cuvette for collecting the extract. At the upper side part of the separator is 1/4" NPT connection, which leads to a flow meter. In the extraction vessel plug, the filter element is placed in order to filter particles 2 µm nominal and 10 µm absolute (Norman Ultraporous 4202T-6T-2M). HPS tubes are of dimension 10×2 mm and are connected to each other by Ermeto couplings (flat, knees, tees). Used high pressure valves were provided by the same company that produces Ermeto couplings (model B-HV). Pressure in the extraction cell is controlled by two WIKA manometers (model 212.20) 60 MPa and one WIKA manometer (model 212.20) 4 MPa for pressure in the separator. CO_2 flow is controlled with Matheson FM-1050 (E800) flow meter. The maximum flow rate that a given flow meter can measure is 63,03 SLPM. A pump used for pressurizing liquid CO_2 is Haskel® MS-71. Liquid CO2 is precooled through SS coil at temperature -18 °C cooled by ethylene glycol/ethanol cooling bath. The pump has the ability to pressure liquid up to 60 MPa. The maximum working pressure is 40 MPa. Then, a pump check valve is located to prevent eventually disorders of CO_2 flow. Finally, the extraction vessel high pressure is reduced by high pressure valve (B-HV) to the desirable pressure (1,5 MPa) leading to the separator.

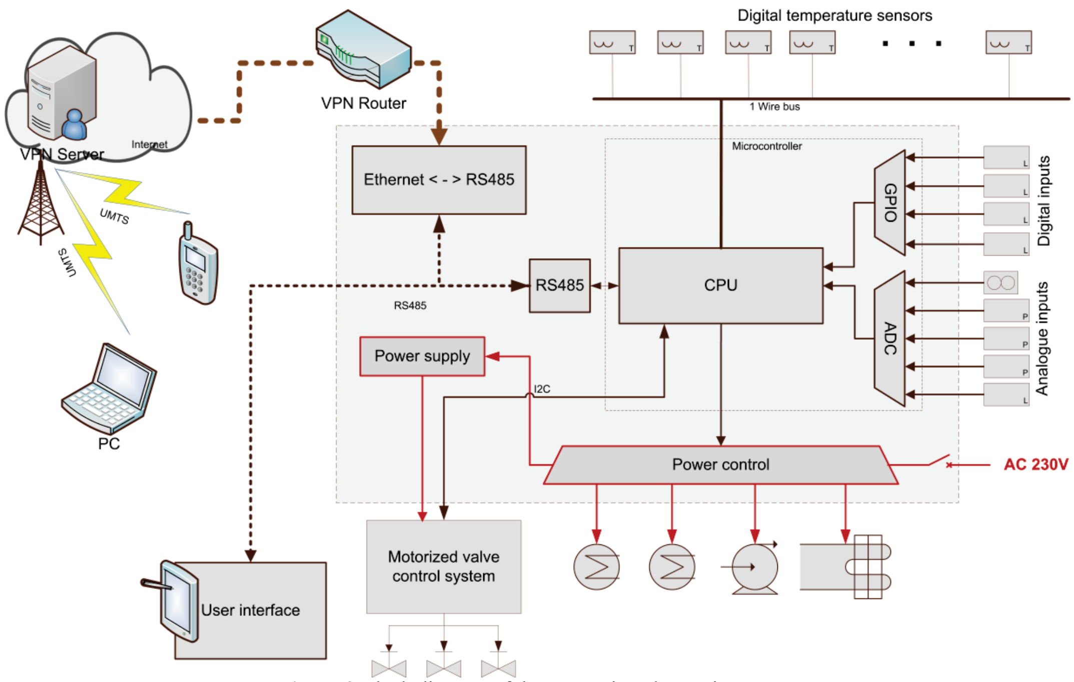

Figure 2 Block diagram of the supporting electronic system

3 Monitoring and controlling the SFE process

Supercritical fluid extraction is a complex process, with large number of variables that needs to be controlled in order to achieve the quality extract. As the process of industrial control is often performed by the use of industrial PLC (Programmable Logic Controllers), the proposed approach in this paper eliminates the need for PLC and offers a cost effective solution for controlling the process of SFE in handmade systems. As depicted in Fig. 1, the supercritical extraction system is composed of various mechanical components with secondary systems that provide the required conditions for the supercritical extraction.

In order to provide the required condition, the supporting electronic system is required for monitoring and controlling the required conditions, thus regulating the process of supercritical fluid extraction. The supporting electronic system is composed of several components (depicted in Fig. 2). The main components can be divided into the following categories:

- Data acquisition,
- Regulation and process control,
- Actuator and power control,
- Communication and
- User interface.

3.1 Process monitoring and sensing

Data acquisition system is composed of hardware and software components designed to present values from the real world in a discrete manner applicable for processing in digital systems. This process is called Analog to Digital Conversion (ADC) and it represents the basic approach in digital data representation. In the HM-SFE system, various physical values need to be digitally represented in order to monitor the flow of the extraction. The main parameters required for the monitoring of the SFE process include multiple temperatures, pressures and flow values that are normally measured using a mechanical instrument (gauge). However, for the digital signal representation this approach cannot be adapted. Instead, a specially designed transmitter needs to be installed for the purpose of measuring the desired values. The most common type of a pressure transmitter is an analogue transmitter that exhibits a linear transfer function described as:

$$P_{rc} = b + aU_m \quad (1)$$

where P_{rc} is the pressure that corresponds to the voltage U_m, a is the slope of the fitted curve and b is its intercept [11].

From the aforementioned, the process of acquiring the pressure data from the environment is pretty straightforward and the following step after ACD is data sampling. Due to the fast dynamics of the process, the pressure values need to be sampled at a much higher sampling rate than other values within the SFE system. Due to the fact that the most dynamic pressure component does not exceed 2 oscillations per second, the sampling frequency of the pressure sensors was chosen to be 14 S/s, obeying the Nyquist-Shannon sampling limit and assuring the data oversampling.

However, due to the superimposed high frequency noise, resulting from other electronic component and the dynamic of the process itself, the data must be filtered using a low-pass filter. To simplify the filtering process, the proposed system uses a simple first order low-pass digital recursive filter, implemented within the micro-processor. The equation in the recursive form defines a single pole low-pass filter. This can be seen from Eq. (2):

$$Y[k] = a_0 \cdot X[k] + b_1 \cdot Y[k-1] \quad (2)$$

where $X[k]$ and $Y[k]$ are input and output values of the sample k, $Y[k-1]$ is the output value of the previous sample $Y[k-1]$, and a_0 and b_1 are weight coefficients. In order to calculate weighting coefficients, two simple equations are required [12].

$$a_0 = 1 - x; \quad b_1 = x; \quad x = \mathrm{e}^{-\frac{1}{d}} = \mathrm{e}^{-2\pi f c} \quad (3)$$

The characteristics of the used filter are controlled by the parameter $x \in [0, 1]$ that represents the amount of decay between adjacent samples. The value for x can be calculated from the desired time constant of the filter, where the time constant d represents the number of samples required for the signal to decay 36,8 % of its final value and f_C represents the cutoff frequency (as a fraction of the sampling frequency). If the cutoff frequency is set at the most dynamic pressure component, f_C results in a value of 0,14, giving the value of weighting coefficients:

$$a_0 = 1 - \mathrm{e}^{-2\pi \cdot 0{,}36} = 0{,}59; \; b_1 = \mathrm{e}^{-2\pi \cdot 0{,}36} = 0{,}41 \quad (4)$$

By implementing the digital low pass filter, the high frequency noise is minimized and the data is rendered noise free. The calculated value of the measured pressure depends solely on the range of the pressure sensor. With the ADC resolution of 10 bits, for the pressure value of the separator the maximum measured pressure is equal to 34,5 bar, with the maximum resolution of 40 mbar.

Further, various other values can be calculated from the pressure information, one of which being the flow rate of the output gas. The simplest method for measuring the flow rate of a gas is by means of a venturi flow meter (Fig. 3).

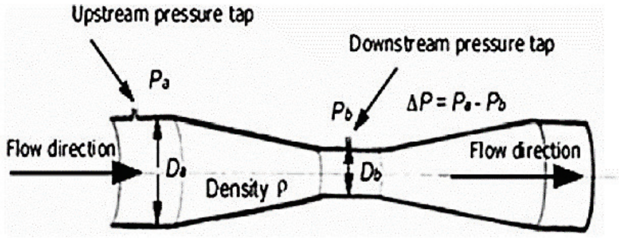

Figure 3 Cross-section of a venturi nozzle [13]

A fluid passing through smoothly varying constrictions experiences changes in velocity and pressure. These changes can be used to measure the flow rate of the fluid. When the fluid reaches the venturi nozzle, the fluid is forced to converge throughout a smaller aperture. The point of maximum convergence occurs at vena-contracta, where the velocity and the pressure changes. The volumetric and mass flow rates can be obtained from the Bernoulli's equation by measuring the difference in fluid pressure between the two stated points [13]:

$$Q = C_d \cdot A_b \sqrt{\frac{1}{(1-\beta^4)}} \sqrt{\frac{2\Delta P}{\rho}} \quad (5)$$

where C_d is the discharge coefficient, A_b is the area of the flow meter cross section, b is the ratio of D_b to D_a, ΔP is the differential pressure and ρ is the density of the fluid. The design of the venturi nozzle was determined from the maximum differential pressure of the pressure sensor (2000 Pa) and the maximum mass flow of the CO_2 gas (5 kg/h). The resulting venturi nozzle was dimensioned at D_a = 10 mm and D_b = 5 mm. When taken into account the resolution of the ADC converter (10 bits), the maximum measured flow is equal to 53 SLPM, with the resolution of 0,05 SLPM.

Due to the fact that the sampling of the analogue data is achieved in a sequence (using a multiplexer and a single ADC), burst errors are evenly distributed along all input parameters. However, sequential sampling of the data in turn affects the overall number of used analogue sensors, primarily due to the restrictions of input multiplexers. Furthermore, with the increased number of the analogue inputs, the sampling frequency also decreases resulting in a lower resolution. The presented drawbacks directly affect the process of measuring temperature values of the process, especially due to the fact that the required number of temperature sensors for the monitoring of the SFE process is greater than 8 sensors.

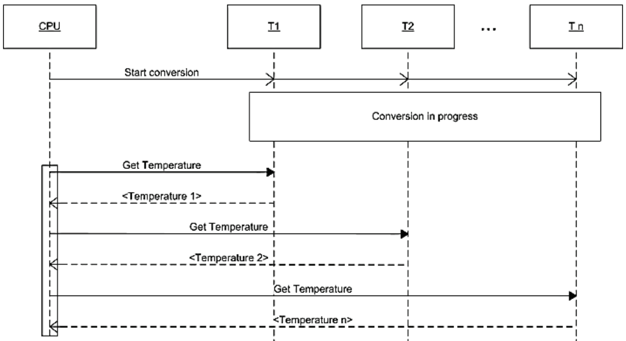

Figure 4 Sequence diagram of the temperature sensors data acquisition

In order to overcome the presented problems, the temperature values were acquired in a different manner. In comparison to the centralized solution for analogue to digital conversion used in the pressure measurement, the temperature measurements use a distributed ADC conversion where each temperature sensor incorporates an analogue to digital converter with an accompanying circuitry (DS18B20 sensor). With the integrated filters, the signal noise is minimized, hence no additional filters are required. However, the consequence of this is seen at the lower sampling rate (1,333 S/s). As the temperature values are slow time changing values, this sampling rate is acceptable for the slow temperature changing process of the SFE process. By using a 1 Wire digital bus system, all sensors can be connected onto a same bus, where the data from a specific sensor is acquired by addressing the sensor with a 64 bit address. To achieve the uniform time sampling, a broadcast message is used signalling the start of the ADC conversion for all sensors on the bus. After the conversion time (750 ms min), the data is collected from each sensor. Communication diagram is shown in Fig. 4. With the integrated analogue to digital converter with 12 bits of resolution, the temperature of the used sensors ranges from −55 °C to +125 °C, with the maximum resolution of 0,0625 °C.

3.2 Controlling the process of SFE

In order to control the actuating components of the system, a transfer from discrete to continuous domain must be performed. The most suitable component for this conversion is a digital to analogue converter that converts quantized and discrete values into analog continuous signals. However, this approach is fairly inadequate primarily because not all actuators can be easily controlled by analogue values. One example is the electric heater control, where the value of alternating current must be varied according to the control signal. This approach is very complex mainly due to the nature of AC power supply. In order to circumvent complex regulation, this paper proposes the use of a solid state relays (SSR) with the ability of AC load control with low voltage DC signals, coupled with low frequency pulse width modulation (PWM). The reason for using low frequency PWM is the fact that the SSR consists from a SCR and an opto-isolator, so once the SCR starts to conduct electrical current, it cannot be switched off until the next half period of the sine wave. By using low frequency PWM (0,15 Hz), whose frequency is much lower than the grid frequency (50 Hz), it is possible to control long RMS value of the current. This approach can only be applied to slow time changing processes, such as temperature control.

On the other hand, for fast time changing processes, such as the control of the pressures and flow, a different approach must be considered. For the control of pressure and flow, the actuating components are the valves, so a conversion from discrete to continuous domain is perceived by using motorized valve control system. The motorized valve control system uses voltage pulses to drive the stepper motors used for the control of the valves. The valves are automatically calibrated at start, so no position sensor is required. By using stepper motors, it is possible to accurately control the position of each valve. The valve control system is controlled by the main microcontroller in a way that each time a new value of the control signal is available from the main microcontroller, the value is transmitted towards the valve control system. In the case of a system failure, a software watchdog timer will act upon all valves and close them to avoid pressure breach.

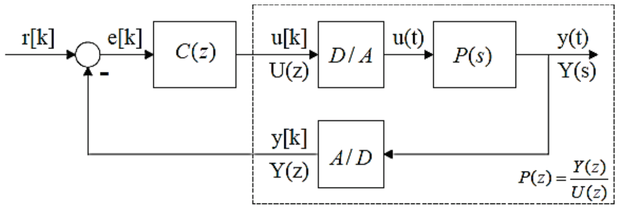

Figure 5 Controlling continues process by discrete regulator

On the side of the process regulation, in HM-SFE system, the temperature of the input carbon dioxide gas must be regulated with precision in order to achieve supercritical state. The proposed system uses two methods of assuring CO_2 gas temperature: the former method utilizes a water heat exchanger, whereas the latter method utilizes an extraction cell heater. Both methods are used simultaneously in order to achieve the desired temperature precision and continuity. The extraction cell is heated with a glass fibre electric heater controlled by supporting electronic system and Solid State Relays (SSR). The temperature control is achieved using the PID regulator with lag delay compensation, due to the large mass of the extraction cell. Temperature measurements and regulation of the extraction cell are performed using an integrated temperature sensor within the cell and additional temperature sensor measuring outputs gas temperature. The input CO_2 line towards the extraction cell is preheated using a heat exchanger, driven by a water heating system. The temperature of the input gas is regulated by measuring three temperatures: water temperature of the water heater, water input temperature in the heat exchanger and the output water temperature. The temperature is regulated using a standard PID regulation taking into account the differential temperatures of water lines and output gas line. A discrete system model that can be easily applied to anymicro-processor unit is shown in Fig. 5 and a discrete PID regulator is shown on Eq. (6).

$$u[k] = K_p e[k] + K_i \sum_{k=0}^{n} e[k] + K_d (y[k] - y[k-1]) \quad (6)$$

Fig. 7 shows the situation where the application (slave 2) assigned to the timeslot 2 sends a write request to the master. The transmission starts after the first keep-alive (KA) sequence on the graph, where the application transmits the write request within the specified second time slot, assigning a new UUID to the master. The success of the sent write command is verified at the second KA packet and it's UUID. On the other hand, in Fig. 8 the process of sending a write command from the user interface system (slave 1) assigned to the first time slot is depicted. As seen from the additional trace on the graph (trace 4), the sent data from the graphical user system is sent through the RS485 bus on the data receive line on the master. As seen, the communication is achieved within the first time slot. Finally, in the case of multiple accesses (Fig. 9), the collision at the RS485 line is avoided by using TDMA and if the received packet conflicts, the first time slot has priority over all other time slots. As seen from the figures, it is evident that the designed protocol was implemented as specified in the protocol description.

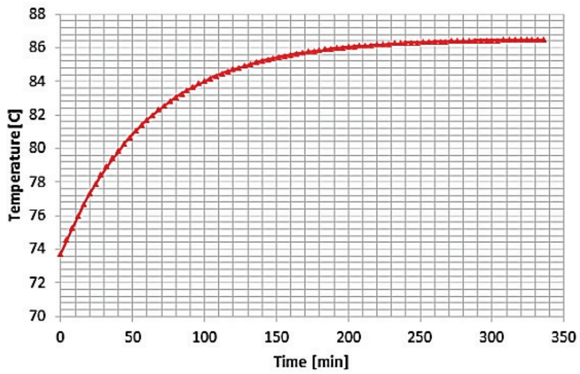

Figure 6 Heat exchanger process reaction curve for control signal step change (control step response 39 → 54)

The resulting value of the PID regulator is transmitted to the actuating modules that exhibit the functionality of digital to analogue converter (DAC) in order to control continuous processes in real life. The parameters for the PID regulator are tuned using the Ziegler-Nichols Open-Loop Tuning Method (Process Reaction Method) by means of monitoring the process reaction curve. The process reaction curve for the temperature control of the heat exchanger is shown in Fig. 6; for control signal step change from 39 to 54 (of maximum 255 steps).

3.3 The concept of integration with Internet of Things

The concept processing equipment integration (such as the SFE system) with the of Internet of Things (IoT) presents a leap forward in the new information era. The concept of Internet of Things relay on the idea of interconnecting various devices and services using a global Internet networks (Fig. 7). As IoT evolves, various aspects need to be addressed in interconnecting new devices and services. The aspect of privacy and security presents an important issue in the area of remote food process control. As SFE systems are used in research and development of new products, the extraction is often performed on small samples. As the extraction can be time consuming effort (up to 8 hours) the integration with IoT enables remote supervision of the extraction, enabling a semi-autonomous operation. However, alongside the advantages there are numerous questions that arise in the IoT integration.

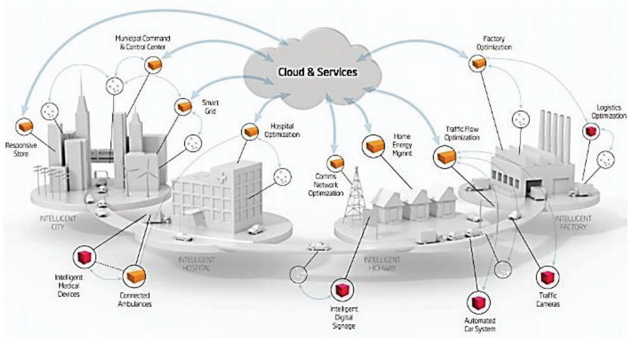

Figure 7 The concept of Internet of Things [19]

As supercritical fluid extraction is based on high pressure gasses operated at medium to high temperatures, the security becomes of outmost importance. When discussing security aspects of IoT and SFE system one must take into account the nature of Internet as a global network. Due to the large number of potential malicious users on the Internet and possible hazardous operations of SFE system this interconnection must be carefully designed. First and foremost the system must be designed to prevent hazardous conditions that can occur upon malicious use. This includes the ability to prevent high pressures and high temperatures that can cause critical design conditions and ruptures of the high pressure systems. In SFE systems high pressures are needed to perform the extraction, however, if the pressures exceed the maximum safety value the extractor can breach, causing CO_2 leakage in the best case or an explosion in the worst case. Also, this can be propelled by high temperatures that weaken the O-ring seals and cause CO_2 leakages at connections. This can be prevented by process control of the system, where maximum pressure/ temperature values are predefined and a mechanical design, mechanically limiting pressure by a safety valve.

As described, security risks can be mitigated on the physical layer of the system, however the problem of data privacy and tampering with the process presents a valid problem. The change of an extraction parameter by a malicious user can cause an error in measurement or a destruction of a sample. Furthermore, the extraction process can be stopped, wasting valuable time of a long running extraction.

Finally, from a security related point of view, a very common denial of service DoS attack must be also analysed. Whether a severed network connection between SFE system and Internet is due to the attack or due to common network equipment failure an end user can be left without supervision of the extraction process. In these scenarios a redundant communication channel must be assured in the form of cellular or satellite connection. Overall, aspects of IoT integration cannot be ignored and must be analyzed in details, especially with the evolution of IoT to Industry 4.0. As a hot and a demanding topic this analysis will be left for future work.

On the other hand, to enable IoT integration the main prerequisite is to enable the exchange of process data and parameters with other systems, such as the user interface (used to control the supporting electronic system) and the Internet in general. In this scope, the communication segment is responsible for the exchange of instantaneous process data and the acquisition of process control data (such as reference temperatures, pressures flow and other regulator references). In addition, the control of the process of supercritical extraction is also enabled by means of starting and stopping the process remotely.

The communication is a time division multiple access system (TDMA) that assigns multiple time slots for multiple access on the RS485 bus. The communication is master oriented where the supporting electronic system acts as a communication master and sets the appropriate time slots. The communication is established in a form of synchronous request/response communication. In order to enable synchronous communication, the master periodically transmits keep-alive package containing a device address, current process values, UUID (Universally Unique IDentifier) and a checksum. The structure of a packet is shown as follows, where the packet header and footer are bolded:

:40R045,15,25,20,30,30,4616,4757,4124,4005,3754,127,20,145,57,2874,151,254,154,47,87,1855,0000000000000000000000000000000**16**

Process values contained within the data packet are coma delimited and the last parameter represents a sequence UUID (128 bit number). The header is composed of a frame start delimiter (:), a device address (40) and a packet type (R0 "keep-alive" packet). End of a packet is delimited by newline (0×0d 0×0a) escape characters. The keep-alive packet is periodically sent every 1 second and the period in between is divided into four time slots. This can be seen from the sequence diagram shown in Fig. 8.

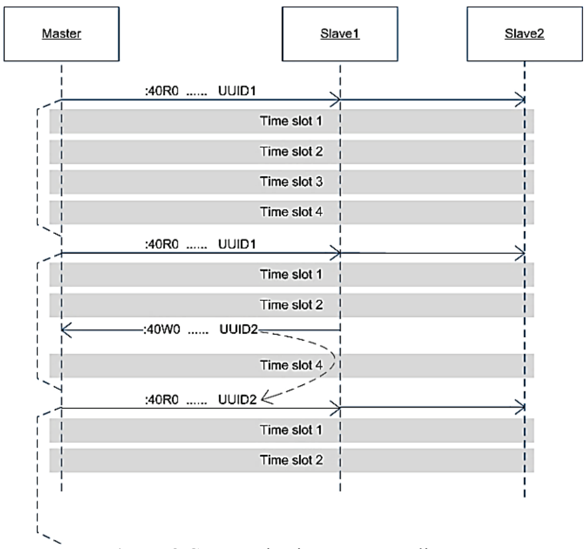

Figure 8 Communication sequence diagram

The advantage of using UUID is the ability to uniquely identify each sequence when a change occurs [14]. For instance, (Fig. 8) in the second sequence, third time slot, a write command is sent from Slave 1 to the master requesting the change of a certain parameter. The Slave device assembling the package checks the UUID from the last sequence (UUID1) and generates a new UUID2 which is appended to the write packet (W0). After the transmission and a successful reception of a write packet, the master acknowledges successful reception and processing of the new packet by updating its UUID value to the value of the last UUID received (UUID2 in Fig. 8). This is reflected on the upcoming keep-alive packet when Slave 1 gets the confirmation of a successful transmission.

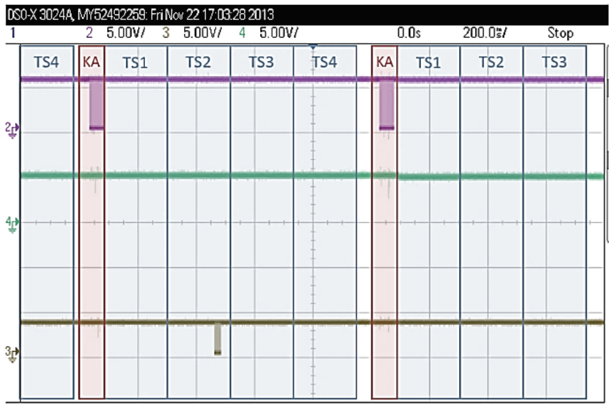

Figure 9 Application (Slave 2) transmits a write request (2 - data transmit from Master, 3- data receive on Master, 4 - data transmit from user interface)

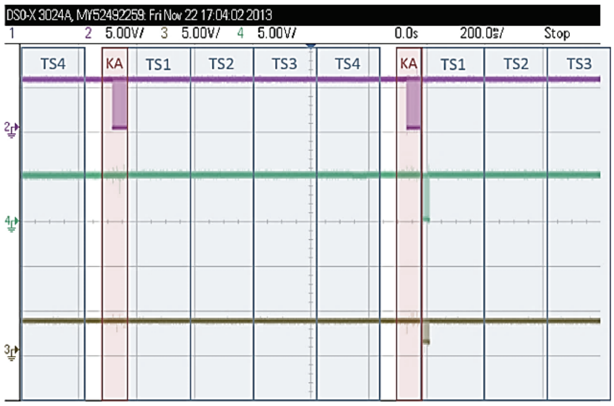

Figure 10 User interface system (Slave 1) transmits a write request (2 - data transmit from Master, 3- data receive on Master, 4 - data transmit from user interface)

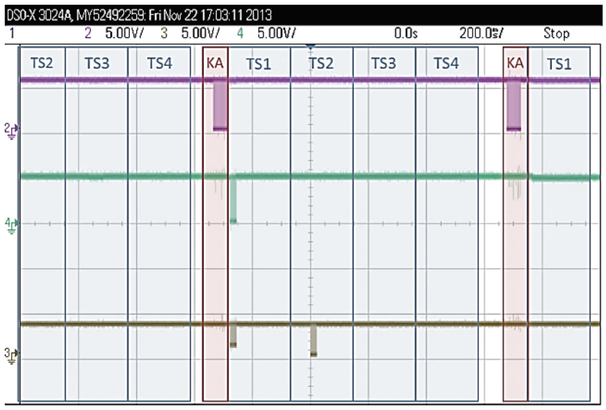

Figure 11 Both application (Slave 2) and user interface system (Slave 1) transmits a write request (2 - data transmit from Master, 3 - data receive on Master, 4 - data transmit from user interface)

Measurement of the described protocol can be seen from Fig. 9-11, where the time slot communication is measured with two slave devices (PC application and a user interface system).

3.4 User interface

User interface is composed of user controls and a liquid crystal display (LCD) used to display process values. The block schematic of the user interface system is shown in Fig. 12. Due to the fact that the communication segment is realized using RS485 communication, it is possible to replace the proposed user interface system with a modern tablet version, with the use of a Bluetooth to RS485 adapter. This approach presents a modular solution that can be adopted to the requirements at hand and to the overall cost of the system.

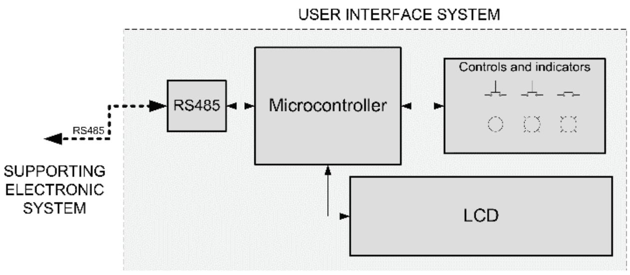

Figure 12 User interface system for SFE device

Finally, an application with the ability for remote controlling and processing data collection was developed for a personal computer. The application was developed in C# running on Windows platform. The application is shown in Fig. 13.

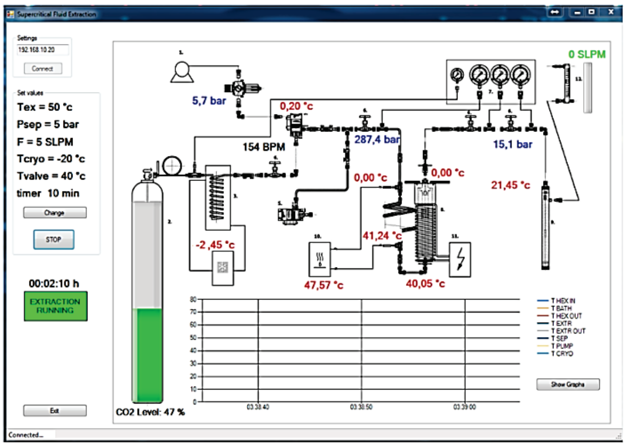

Figure 13 Developed PC application for the HM-SFE system

4 Economic evaluation

Most scientists believe that SFE technology is very expensive due to very high investment costs in comparison with classical low-pressure equipment, or other equipment for extraction. Reliable cost estimation of SFE equipment is not presently available from published sources, and the prices can drastically change according to the type of equipment, instrumentation automation, etc. The SFE equipment consists of basic components such as: compressor or high-pressure liquid pump, extraction vessel, separator, CO_2 tank, several heat exchangers followed with related piping, valves, instruments, utility services required for a reliable and safe operation in automatic mode.

Perrut [20] in his work gave cost estimation for the various types of applications, based on his experience of equipment supplier and SFE plant operators. He had reported that all prices for such unit (represented by a dimensionless price index *PI* on a logarithmic scale) are near to a straight line with a slope of 0,24 versus the log of product of total volume V_T by the design flow rate Q (Eq. (7)):

$$PI = A \cdot (10 \cdot V_T \cdot Q)^{0,24} \quad (7)$$

Perrut [20] shows also that capital amortization sharply decreases when capacity increases, what is a strong incentive to use large capacity multi-products units in "time-sharing" rather than operating small capacity units dedicated to only one product, when possible!

In this work we present the data we have gathered from our own experience of building laboratory SFE plant. So the equipment cost for our handmade SFE system was 12 500 € (EURO)!

The variable costs gather energy and fluids (carbon dioxide). On small and medium-scale units, energy is not so expensive, especially when heat is supplied by steam available on site or hot water heated by fuel or gas; electrical heating is the most flexible but should be limited to small-scale units like our handmade SFE equipment. For very large-scale units it may be valuable to use a recompression unit to recover this CO_2 and recycle it. Of course, pressure vessels must be inspected and submitted to pressure tests according to official standards. Moreover, the main process valves must be often checked as they are the key of safe operation during opening for raw material change. Sensors must be recalibrated periodically, in comparison with traceable reference sensors, and data logging validated.

5 Comparison of the obtained extraction oil yields at the handmade system with the commercial SFE system

The results obtained with the HM-SFE system were compared with the commercial SFE system (HPEP, NOVA-Swiss, Effertikon, Switzerland). The obtained extraction oil yields were monitored in both systems (handmade and commercial SFE system). The commercial laboratory-scale high pressure extraction plant has been explained in detail elsewhere [15]. 130 g of ground soybeans was placed into the extractor basket in each experiment. The extraction conditions in both experiments were fixed at the pressure of 40 MPa, temperature of 40 °C and CO_2 flow of 0,476 kg/h. The extracts were collected in glass tubes previously weighed and placed in the separator at ambient temperature and pressure. The amount of extract obtained at regular intervals of time was established by weighing (balance precision ±0,00001 g). The separator conditions were 15 bar and 25 °C. The accumulated product samples were collected and weighed hourly. The extraction was carried until the amount of the product sample decreased below 0,1 % mass of the raw material. Commercial CO_2 (Messer, Novi Sad, Serbia) was used for supercritical fluid extraction.

The soybean seeds (cultivar "Ika") used for supercritical CO_2 extraction were obtained from the Agricultural Institute Osijek (Croatia). Moisture content was determined by oven drying to constant weight at 105

°C [16] and noted as percentage (11,02 ± 0,11 %). The material was ground and sieved using a vertical vibratory sieve shaker (Labortechnik GmbH, Ilmenau, Germany) for 20 min explained in detail elsewhere [9]. The initial oil content was measured by the traditional laboratory Soxhlet-extraction with n-hexane. 30 g of ground soybeans was extracted with 250 mL solvent until it was totally depleted. The whole process took 16 h. The measurement was done in triplicate. The average oil content in soybeans for three replicates was 20,08 ± 0,14 %. Reagent-grade n-hexane (J. T. Baker, Milan, Italy) was used for the laboratory Soxhlet-extraction.

The obtained extraction yield at newly designed HM-SFE system is given in Fig. 14 and the results were compared with the extraction yield obtained at the commercial SFE system which was published in detail in our previous paper [9]. It can be concluded that the obtained extraction yields in the HM-SFE system are very similar to those obtained by the commercial SFE system. Hence, it can be concluded that the HM-SFE system permits to obtain extracts in a cost effective way.

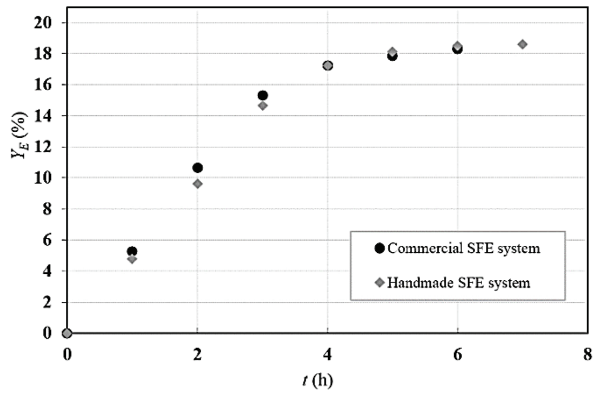

Figure 14 Extraction yield of soybean oil obtained by supercritical CO_2 at the commercial and HM-SFE system. Conditions: T_E = 40 °C; P_E = 40 MPa; m_f = 0,476 kg/h

6 Conclusion

This paper presents a design approach of a handmade supercritical fluid extraction system used as a small scale research SFE system. In the process of the design, mechanical design and development are performed, followed by the implementation of an electronic process control system and implementing a remote supervision and control system with the ability of presenting relevant data to an end user. Upon the completion of the system design, the comparison of the proposed system is drawn in respect to the existing commercial system. The measurements included extraction yield during a period of time for the two systems. From the measurement results, it can be concluded that the statistical error between the compared systems is less than 10 %, representing a significant correlation of the data and the validation of the proposed HM-SFE system.

The proposed system offers a cost effective solution for small scale research SFE systems with the ability of detailed parametric analysis and remote process supervision, normally not available in industrial grade SFE systems. The fact that the system enables remote parameter supervision and remote process control presents an advantage for scientific research. Additionally, using the precision temperature control, a supercritical state of the CO_2 can be guaranteed eliminating the possibility of not achieving supercritical conditions of the fluid.

Finally, future work on this topic includes detailed analysis on the concepts of integration of SFE system with IoT. While this paper gives a general overview of the integration concept an additional and more detailed analysis should be performed, particularly from the security aspect. As SFE systems are based on high pressure that can result in hazardous conditions, it is of outmost importance that IoT integration follows guidelines for achieving maximum security and avoiding the possible hazardous conditions even in cases of security breaches. With the IoT expansion to Industry 4.0 the proposed integration of similar systems is becoming a hot research topic in scientific communities, which propels the need for continuous and interdisciplinary approach for solving the open issues of the field.

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