



Project co-financed by the European Union from the European Regional Development Fund

**Paulina ŁOBODZIN, Marian GRĄDKOWSKI**

Institute for Sustainable Technologies National Research Institute in Radom  
paulina.lobodzin@itee.radom.pl, marian.gradkowski@itee.radom.pl

## **TESTING MICROFILTRATION INSTALLATION FOR AQUEOUS OPERATING FLUID PURIFICATION**

### **Key words**

Ceramic membrane, back pulse, pilot plant.

### **Abstract**

A model of microfiltration installation for regeneration of aqueous operating fluids was created and built. The monitoring and measurement system was installed and all components were calibrated. The correctness of the construction and functionality of the installation was verified. As a part of the verification of monitoring and measurement system, a series of simulated filtration processes was conducted. The authors examined the activation of the pump and „back pulse” procedures, the security and sensor response to parameter changes, and the quality of the hydraulic connections. It was found that the monitoring and measurement system of the membrane installation works properly and it is ready to work in the laboratory and semitechnical tests using real contaminated aqueous operating fluids.

### **Introduction**

For some time, increasing demands on water quality for industries and especially for the consumer is observed. It is connected with shrinking resources of good quality water and the growing costs of its acquisition. Against this background, legislation in the field of water management is moving towards

intensified protection of its resources. An example of such activities is the framework for Community action in the field of water policy adopted in the EU countries. Its aim is to improve the protection of waters through the introduction a common European water policy, based on a transparent effective and coherent legislative framework. It obligates the EU Member States to the rational use and protection of water resources in accordance with the principles of sustainable development [1]. It calls for the reduction of water consumption in manufacturing processes and the increase in the quality of water after these processes, because the water is discharged into sewage system or natural receives. All those factors are forcing engineers to design processes with closed water circuits, where possible. These factors have become the essential causes of the search for new, effective methods for water treatment after manufacturing processes.

Conventional methods used for water treatment often involve complex technological lines that consume large amounts of energy and chemicals. Despite such combined processes, water treatment is not always effective enough [2]. Frequently, it leads to the generation of new waste that is difficult to manage. Pressure membrane technologies proved to be an interesting alternative to conventional water treatment. They have been used in almost every field of science and technology. The advantages of membrane processes, in addition to lower energy consumption or easy scale-up by multiple membrane modules, are that the waste stream only contains the impurities removed from the water without any by-products of chemical reactions or other substances released during the water purification. The process of separation of contaminants takes place in a purely physical way, i.e. components do not undergo physical, chemical, or biological changes. In the case of wastewater, properly selected membrane techniques enable almost complete purification of water and concentration of contaminants, which often facilitates their subsequent recycling with other technologies. Commonly used in water and wastewater treatment are all pressure membrane techniques, particularly microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) [3].

When purifying solutions containing large amounts of dispersed contaminants, the best results are obtained in the case of microfiltration. This case belongs to the low-pressure membrane processes in which, depending on the cut-off of the membrane used, the size of separated particles is in the range 0.1–20  $\mu\text{m}$ . Therefore, the resulting solution contains only those particles that are smaller than the cut-off of the membrane, as well as soluble substances. The separation mechanism is based on the sieve action of the porous membrane. The process enables the separation of two-phase systems (emulsions and suspensions) and the secretion of high molecular substances, for example, polysaccharides, colloids with highly developed hydration shell, and certain proteins, in the concentrate leaving the finest particles, colloidal substances, low molecular substances and also salt [4]. Therefore, microfiltration is predestined

for the regeneration of soluble low molecular weight substances, such as much of aqueous operating fluids.

The aim of this work was to build a pilot microfiltration plant for laboratory and semi-commercial application that would be suitable for a variety of aqueous liquid technology processes.

## 1. Experiment

Microfiltration systems can differ in structure and the way the process parameters and the installation control system are monitored. Generally, membrane systems can be used as manual, semi-automatic, and automatic monitoring and measurement systems. In the first case, process parameters are measured using analogue meters, and the operating parameters are set manually. The opposite of this method is the automatic control system, in which the measurement signals are transferred to the controller (PLC or computer) that supervises process parameters or the operation of the system. Intermediate solutions are semi-automatic systems, in which the repetitive processes are controlled systemically (automatically), while the parameters are often changed, and those that do not have efficient algorithms are controlled manually. This type of a system is a hybrid system, and it is chosen mainly for economic reasons [5, 6].

A microfiltration pilot plant was built for the regeneration of aqueous operating fluids. Due to the experimental nature of the installation, the hybrid measurement and control system was designed. The monitoring and measurement system should be flexible enough to be easily implemented in devices with similar functionality but different structures. A properly designed membrane system should enable continuous measurement of at least the following:

- Fluid pressure at the inlet and outlet of the filter modules, which allows one to determine the operating and transmembrane pressure; and,
- Retentate and permeate flow, which makes it possible to determine the amounts of the product (filtrate), concentrate, and the total amount of the processed liquid with the knowledge of the operating pressure [5, 6].

When building the model of the prototype installation for microfiltration, the authors assumed that it would be operated primarily in the laboratory for experimental treatment of waste (contaminated) water operating fluids, delivered by a potential user of the system. In addition, it was also assumed, that the system could be also tested by the potential end user. With this purpose of the plant it is not feasible to design the purification of liquids with known characteristics and a known range of variation of parameters characterizing it. Therefore, it was established that the properties of the fluid can vary over a wide range, and that abrasive particles, dispersed oils, chemicals, microorganisms, and

a number of other ingredients can occur. It should be understood that the pH of the liquids can be strongly acidic as well as basic. It was also planned that the liquid will be supplied in 1000 dm<sup>3</sup> „mauser” tanks. Therefore, the surface of the membrane and the point of operation of the system were set in a way enabling such purification of the liquid within one work cycle, including maintenance. Considering all these factors, a more expensive but more durable „star-step” ceramic membranes made of a chemically resistant mixture of oxides of aluminium, titanium, and zirconium were used. The filter module was equipped with six 19-channel membranes, with the length of 1200 mm, 0.2 µm cut-off, and 0.33 m<sup>2</sup> of the filtration area. The membranes were placed in a steel cylinder connected with the hydraulic system as shown in Fig. 1.

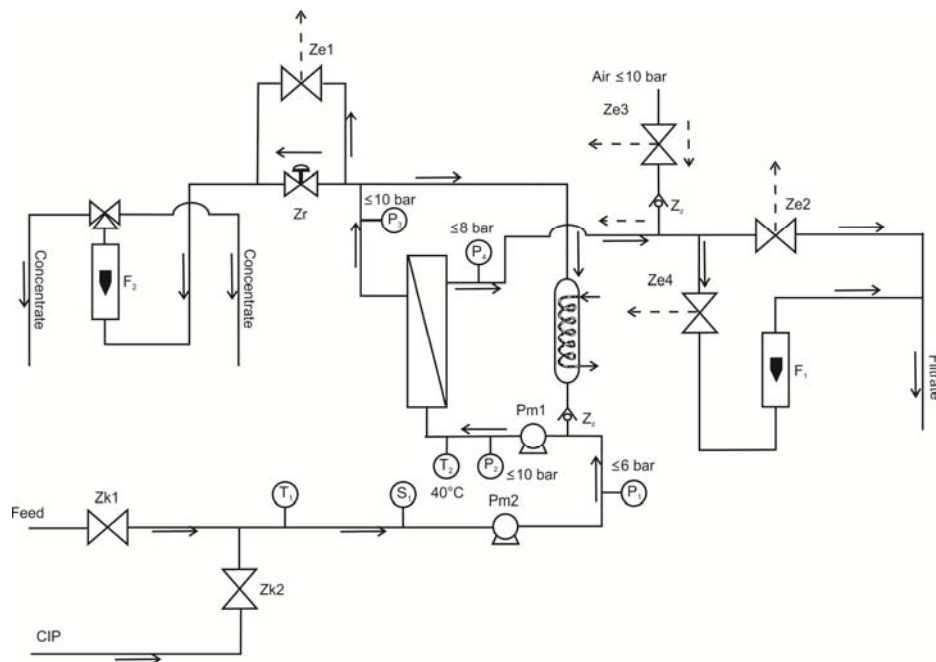


Fig. 1. Hydraulic diagram of the microfiltration plant with the indicated places to locate valves and sensors (PID)

The intended use of the plant as well as potential areas of application determined the structure and functionality of the installation (Fig. 2) and monitoring and measurement system. First of all, the installation had to be mobile, lightweight, and easy to transport by vehicles commonly used in companies. Therefore, its size was adjusted to the open load-carrying body. The device was equipped with wheels, which increased its mobility, and the construction materials were selected in such a way that the weight of <100 kg would be guaranteed. Due to the high chemical resistance of ceramic membranes used, they are located in a module made of stainless steel. In contrast,

the pipelines were made of chemically resistant, but lightweight PVC, combined with an anaerobic adhesive. Because PVC pipelines are used, the temperature of operation of the system is limited to 60°C. The prototype character of the installation enabled the cancellation of many sensors measuring the physicochemical characteristics of regenerated liquids, such as densitometers, turbidity meters, conductivity meters, or colour meters. All these measurements can be successfully performed in the laboratory. Because of the anticipated nature of the installation, it was impossible to pre-programme the parameters of its operation. However, independent adjustment of flow and pressure in individual lines was possible in a relatively wide range. In view of costs, convenience, and the ease of readouts, the installation was equipped with analogue sensing and actuator (valves) elements controlled manually.

In order to reduce the weight of the installation, cleaning of the membranes of the filter cake is carried out by the „back pulse” technique with an external source of compressed air. The unchanging nature of this operation enables its implementation in an automatic mode, with the possibility of free programming of the cycle by the user. The automation of this operation mainly stems from its critical importance for the efficiency of the device and also from the possibility of improper membrane cleaning conducted by the potential end user. Since the installation has to meet the requirements of a laboratory device, some additional elements (e.g. temperature meters, heat exchanges, etc.), which normally are not needed in actual industrial applications, were included. The installation can operate in a batch or continuous mode.



Fig. 2. A general view of membrane microfiltration plant designed to test the regeneration of aqueous operating fluids

In the „batch” system, the installation works in the following rhythm. The process starts with filling the operating fluid (feed) tank. After the connection of the tank and initiation of the installation, the content of the tank is forced through the membrane module. The filtrate (permeate) is drained out, the volume of the fluid in the tank decreases over time, and the concentration of contaminants comes after. The purified liquid circulates between the tank and the installation to reach the critical density of contaminants in the feed tank, above which further filtering is inefficient.

The feed and the circulation pumps are controlled by the monitoring-measurement system installed (Fig. 1). The signal to run both pumps in a sequence (the feed pump and, after a certain time, the circulation pump) is fed from the dry run vibration sensor to prevent seizure. The control system also monitors the course of the „back pulse” operation, whose purpose is to treat the active surface of the membrane filter and to provide the optimum flow filtration. Because of the need to adjust the hydraulic conditions of the installation to the characteristics of the media to be regenerated, flows and pressures in the individual elements of the installation are controlled and steered manually. This makes it easy to determine the most favourable parameters of the process.

All electrical and electronic components (programmable controllers, power supplies, contactors, etc.) are installed in a control cabinet (Fig. 3), which is located on a support frame of the microfiltration installation, above the potential level of the waterspout. After verifying the connections, the sensors were calibrated, and the times of individual installation sequences were set using a PLC.



Fig. 3. A general view and the interior of the control cabinet: 1 – 24 V power supply, 2 – motor protection – circuit breaker PKZ-type, 3 – PLC 819-AC-RC 115/240 V / AC, 4 – motor contact

After the installation of the monitoring and measurement system, its test run was conducted to adjust the output parameters of the installation. For that, tap water with a hardness of 10.22 mg (content of  $C_aCO_3$  in 1 litre of water) was used as the medium. This allowed the verification of basic parameters of

operation and the efficiency of the installed monitoring-measurement system. During these experiments, the tightness of the hydraulic system was tested and the necessary corrections were made. In tests, the feed was fed to the membrane module using a circulation pump (PM1, Fig. 1), operated at a constant flow rate, supplied with feed pump (PM2), and operated at the pressure of 2.0 bars. The filtrate and the concentrate flows were controlled by rotameters F1 and F2. During the pressing of the feed to the membrane module, the permeate stream of the filtrate and its temperature were measured. Both measurement instruments were placed on the wire outside the membrane module.

After adjusting the parameters, the reliability and stability of the system were investigated for the installation. For that purpose, the device worked for 14 days in the closed mode in which the permeate and retentate were not removed. At the same time, the increase in the temperature of the medium used was investigated in the closed mode without the liquid being cooled down. Simultaneously, changes in the viscosity of the medium, the filtration flux (permeate), as a function of temperature, were measured. The relation between the liquid temperature and the flow rate of the filtrate through the membrane (stream) is shown in Fig. 4, and filtration flux changes with changes in viscosity are shown in Fig. 5. Knowledge of these relations will help during the selection of operating parameters for the regeneration of actual contaminated aqueous operating fluids.

As expected, due to the transfer of energy to the system by a pump and external and internal fluid friction, during the circulation in a closed mode (no mass exchange with the surroundings), the temperature of the liquid increased with circulation time (Fig. 4). Correlation coefficient tests showed that the second-degree polynomial  $R^2 \approx 1$  perfectly describes the temperature increase in time. However, from the point of view of control, the most convenient is linear dependency. A high value of the Pearson coefficient for the proportional liquid temperature increase over time of almost 0.96 indicates that, with sufficient accuracy, this dependency can be used to control the installation. More important, however, is the influence of temperature on fluid flux; with the increase in temperature, the fluid flux increases as well, which is connected with the drop in the viscosity of the liquid. In this case, the correlation between the temperature of the liquid and the filtration flux is also very well described by a polynomial of the second degree and sufficiently by linear relation. The increase in temperature of the liquid observed during the 7-hour test can be considered moderate, indirectly indicating the correctness of the selection of pumps and hydraulic system, and particularly, the proper selection of a membrane and the conditions of its operation.

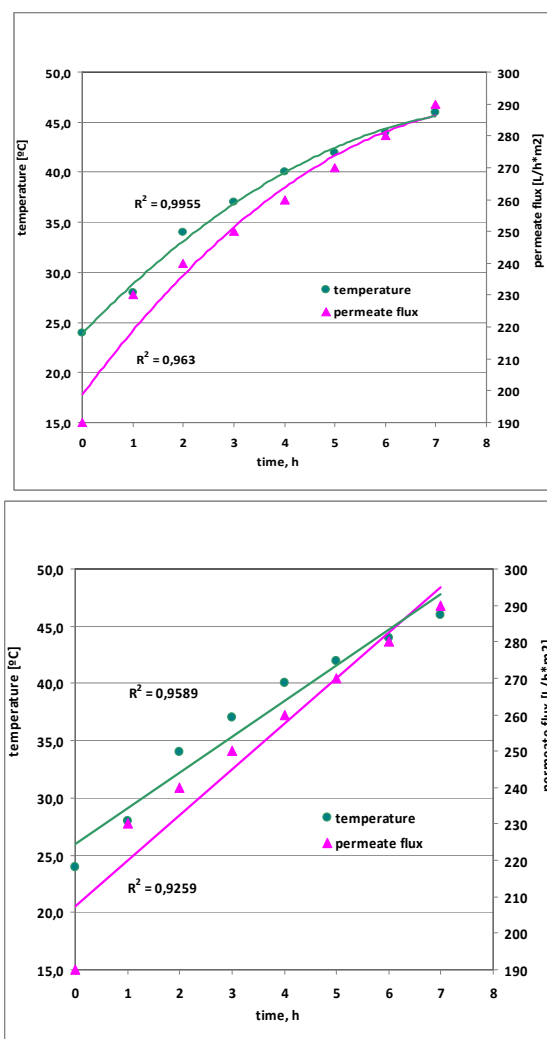


Fig. 4. The increase in temperature and filtration flux during the tests with recirculation of permeate and retentate

The fact that the fluid viscosity decreases with the increase in temperature is important for filtration. The lower viscosity can get higher streams of filtration. Frequently though, the efficiency of purification decreases at the same time as well. It also happens that for greater flux, the micropores are blocked much quicker, and the surface of the membrane needs to be cleaned far more often [4]. Therefore, it is important to know the relationship between the viscosity of the filtered liquid and the permeate stream. As shown at Fig. 5, the reduction of liquid viscosity, associated with the increase in its temperature, accompanies the second-degree polynomial described and the linear increase



in the flux. The increase in the filtration flux is not proportional to the reduction of viscosity, but it is faster than a decrease in viscosity. However, as in the case of the flux, the influence of temperature on the flux of the filtrated liquid and the relationship between the viscosity and the flux can be described with linear equations.

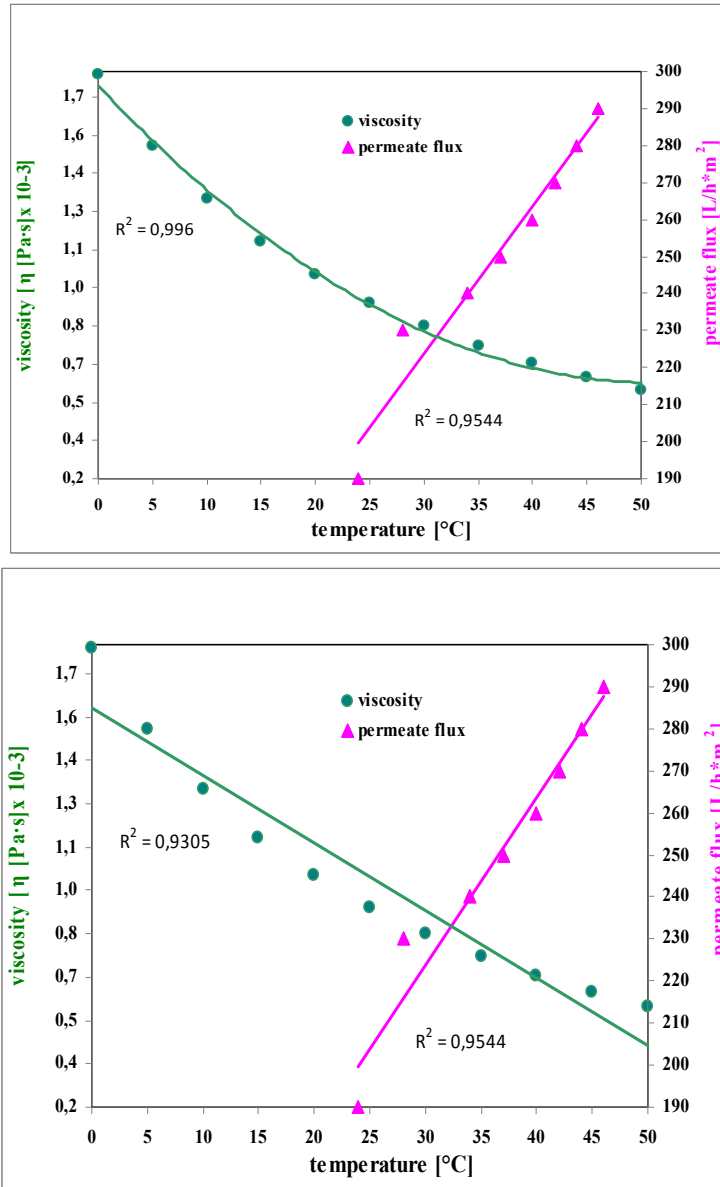


Fig. 5. The dependence of viscosity on temperature and the flow filtration on viscosity of the feed

The initial tests of the model prototype microfiltration installation helped the authors to confirm the correctness of both its structure and the assumed functionality. The authors also stated that the productivity of the permeate can be controlled through linear dependencies between liquid temperature and viscosity. It facilitates the selection of control algorithms and programming drivers.

### **Conclusion**

Membrane installation with microfiltration ceramic membranes was constructed, tested, and verified. The installation is equipped with a monitoring-measurement system with a programmable logic controller (PLC) that ensures correct operation of selected conditions. The tests enabled calibration of the installation and preliminary setting of its operating parameters. It is also possible to conduct laboratory tests and industrial tests. The solutions used enable the replacement of ceramic membranes and the adjustment of their nominal cut-off limit (according to the catalogue card) to given process requirements. Thanks to the use of the PLC, the operating parameters are conformed to the type of membranes used and the quality of the regeneration liquid.

*Scientific work executed within the Strategic Programme “Innovative Systems of Technical Support for Sustainable Development of the Country’s Economy” within Innovative Economy Operational Programme*

*The installation was created in cooperation with the Faculty of Chemical and Process Engineering, Warsaw University of Technology and PolymemTech Sp. z o.o. in Reguły*

### **References**

1. The Water Framework Directive 2000/60/WE from 23.10.2000.
2. Bowen W.R.: Water engineering for the promotion of peace. Desalination and Water Treatment, 2009, 1, 1–6.
3. Van der Bruggen B., Verberk J.O.J.C., Verhack J.: Comparison of pressure-driven membrane processes and traditional processes for drinking water production in Europe based on specific impact criteria. Water SA, 2004, 30, 413–414.
4. Van der Bruggen B., Kim J: The use of nanoparticles in polymeric and ceramic membranes: Review of manufacturing procedures and performance improvement for water treatment. Environmental Pollution, 210, 158, 2335–2349.

5. Kołtuniewicz A.B., Drioli E.: Integrated membrane operation in various industrial sectors, [in:] Comprehensive Membrane Science and Engineering. Elsevier 2010, 4, 109–164.
6. Piątkiewicz W.: Wybrane aspekty projektowania membranowych instalacji filtracyjnych o przepływie krzyżowym. Wydawnictwo Naukowe Instytutu Technologii Eksploatacji – PIB 2012.

### **Testowa instalacja mikrofiltracyjna do oczyszczania wodnych cieczy technologicznych**

#### **Słowa kluczowe:**

Membrany ceramiczne, back pulse, testowa instalacja.

#### **Streszczenie**

Zaprojektowano i zbudowano modelową instalację mikrofiltracyjną przeznaczoną do próbnej regeneracji wodnych cieczy technologicznych. Zainstalowano w niej układ kontrolno-pomiarowy i dokonano niezbędnej kalibracji wszystkich elementów instalacji. Zweryfikowano poprawność konstrukcji oraz funkcjonalność instalacji. W ramach weryfikacji systemu kontrolno-pomiarowego przeprowadzono serię symulowanych procesów filtracji. Podczas nich sprawdzono poprawność zaprogramowanej sekwencji załączania pomp oraz procedury „back pulse”, reakcji czujników i zabezpieczeń na zmiany parametrów, a także jakość połączeń hydraulicznych. Stwierdzono poprawność funkcjonowania systemu kontrolno-pomiarowego instalacji membranowej i potwierdzono jej gotowość do pracy w warunkach testów laboratoryjnych i półtechnicznych z użyciem rzeczywistych, zanieczyszczonych wodnych cieczy technologicznych.