TESTING OF A NANOFIBROUS TEXTILE BY SUBMICRON MONODISPERSE PARTICLES IN A LASER SHEET

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Abstract

This paper deals with testing of a nanofibrous filter by submicron monodisperse polystyrene particles of several sizes. The nanofibrous materials made by the electrospinning technology are very potential as a liquid filters. During the development of new filters it is necessary to determine reliably one of the most important parameter – filter efficiency. In this article the filtration efficiency is measured on the basis of the visualization of a filtration process. A laser sheet is aimed to the place where a filter mounted is. A digital camera records the flow in the laser sheet and by an image analysis the local filtration efficiency is calculated. The experiments were carried out on the water filtration setup including the filtration chamber with an optical access. The bubble point test is used for determination of the maximum, medium and minimum pore size of the filtration textile. The information about pores is compared to the local filtration efficiency and pressure drop of a nanofibrous textile tested by submicron monodisperse particles.

Keywords: Nanofibrous filter, water filtration, flow visualization.

1. INTRODUCTION

Filtration of water is an important process which influences almost every branch of industry. The filtration is performed by several methods, where the most used for drinking water is ultra filtration by fibrous filters, fine sieves and membranes. The nanofibrous filtration textile is a recently developed filter with several benefits compared to the membranes. The nanofibrous membrane is composed from fibers smaller than one micrometer. The most discussed and performed fabrication of nanofibrous filters is an electrospinning. The advantages of the electrospinning lay in its versatile and cheap mass production. The principle is to draw of a fiber by electrostatic forces. For industry the Nanospider method is often used. The nanofibers are electrospinned from a free surface of a polymer solution. The polymer solution represents the positive electrode and a grounded target is the negative electrode [1, 2].

PES/PET filtration material was tested in work of Homaeigohar et al. [3]. The filter was made from a PES nanofibrous net forced on a PET microfibrous support layer. The PES nanofibrous layer was treated by heat because of interfiber adhesion of the individual nanofibers. The filtration material was tested by micron and submicron polystyrene particles (with size of the particles below and above one micron). The retention performance of the filter was determined in several time durations, where the mass concentration of a feed suspension was 0.35 g/liter. A permeate was then examined after each experiment to determine the filtration efficiency. Pressure drop and flow rate were also measured during the experiments. If the size of the feeding particles was closer or higher to the average pore size of the filter, the membrane rejected the particles without significant decrease of flux. But if the size of the particles was lower, the filter was clogged with dramatic rise of a pressure drop. This was caused by packing of the particles together and blocking the pores of the filter. The surface of the nanofibrous layer was covered by a relatively dense cake. The nanofibrous filter demonstrated high efficiency during filtration of micron particles and has a potential to be used in pretreatment applications.

Nanofibres were also used in a food processing to remove bacteria and yeast cells, where their size is from 3 to 12 microns [4]. A dead end filtration during constant flow rate was performed with beer. It was revealed that the yeast in beer forms soft cakes and the pressure drop is not so significant, so the use of nanofibrous materials in beverage processing is promising.

In this article a nanofibrous filter layer supported by a microfibrous layer was tested by submicron monodisperse artificial particles. The experiments were carried out on the water filtration setup including the filtration chamber with an optical access. The filtration process was visualized and the filtration efficiency was determined. The bubble point test was used for determination of the maximum, medium and minimum pore size of the filtration textile. The information about pores was compared to the local filtration efficiency and pressure drop.

2. EXPERIMENTAL SETUP

The filtration process is visualized in the experimental setup, which allows an optical access to the place, where a tested filter is situated. The experimental setup measures pressure drop and flow rate and it is possible to take away samples of water in front of and behind the tested filter. The scheme of the filtration setup is shown in Fig. 1. The flow rate can be set from zero to 10 liters per minute and the pressure drop can reach almost 50 kPa. The inner square shaped cross section of the filtration channel is 5 cm large and the channel is 50 cm long. The flow rate can be controlled on the constant value by a PI controller. The laser sheet is 1 mm thick and 60 mm wide and it is generated by a cylindrical 10° angle Powell lens. Wavelength of the laser beam is 532 nm and the maximum power achieves 98.2 mW. The power was measured by LabMaster Ultima v. 2.35 with mounted attenuator 1:1000. The camera was used Pike 210B/C with lens Samyang 35 mm, F1.4. The resolution of the camera was set to 960 x 540 pixels and images are scaled in 16 bit of grey value and saved in RAW format.

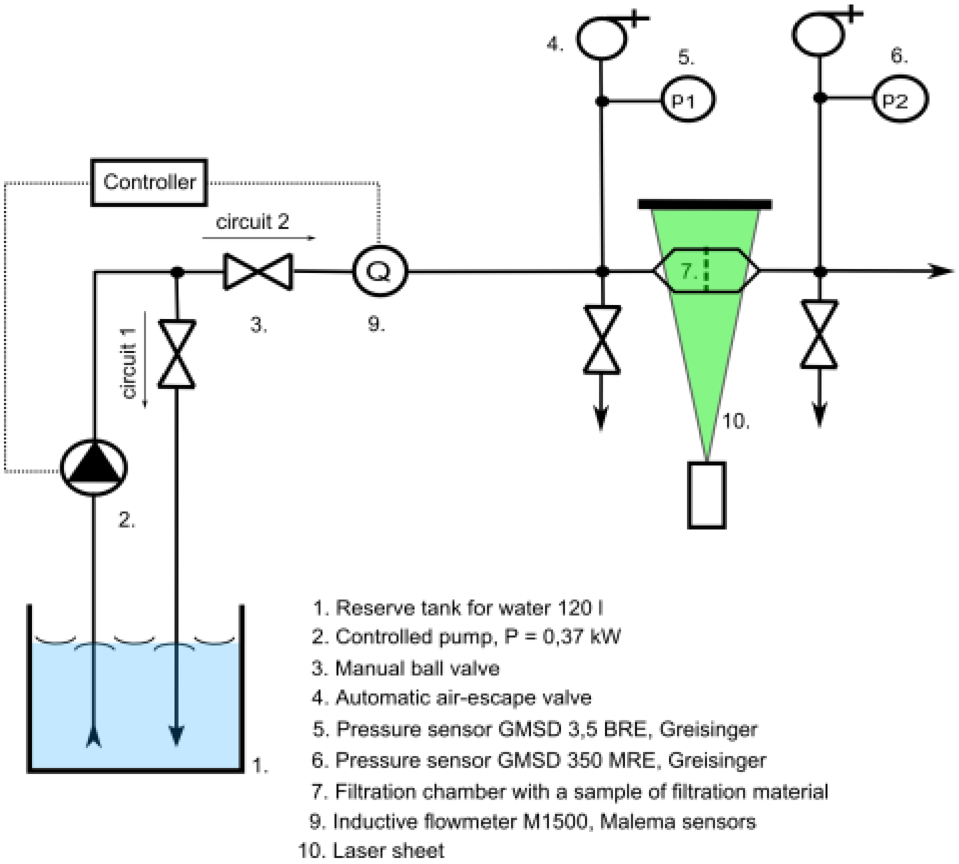

Fig. 1 Scheme of the filtration setup.

3. EXPERIMENTS

3.1. Materials

The local filtration efficiency was measured on the basis of visualization of a filtration process in a laser sheet. Cleaned tap water was seeded by artificial particles 0.28, 0.42 and 0.69 µm large. As the seeding particles the monodisperse polystyrene microspheres obtained from Polysciences Europe GmHb were used. Polystyrene has very optimal optical and mechanical properties, its mass density is 1050 kg/m^2 and relative refractive index of light is 1.5983.

As a tested filter the nanofibrous textile made by the technology electrospinning was used. The electrospinning generates nanofibres by electrostatic forces from a polymer solution. The basis weight of the nanofibrous textile was measured by a precise scale. The material of the nanofibrous textile is polyamid and the basis weight is 0.34 g/m^2. The microfibrous support textile was made of polypropylene. The nanofibrous web was deposited on the support microfibrous polypropylene textile and hot calendered, Fig. 2. Another microfibrous polypropylene textile protects the nanofibrous web from the front.

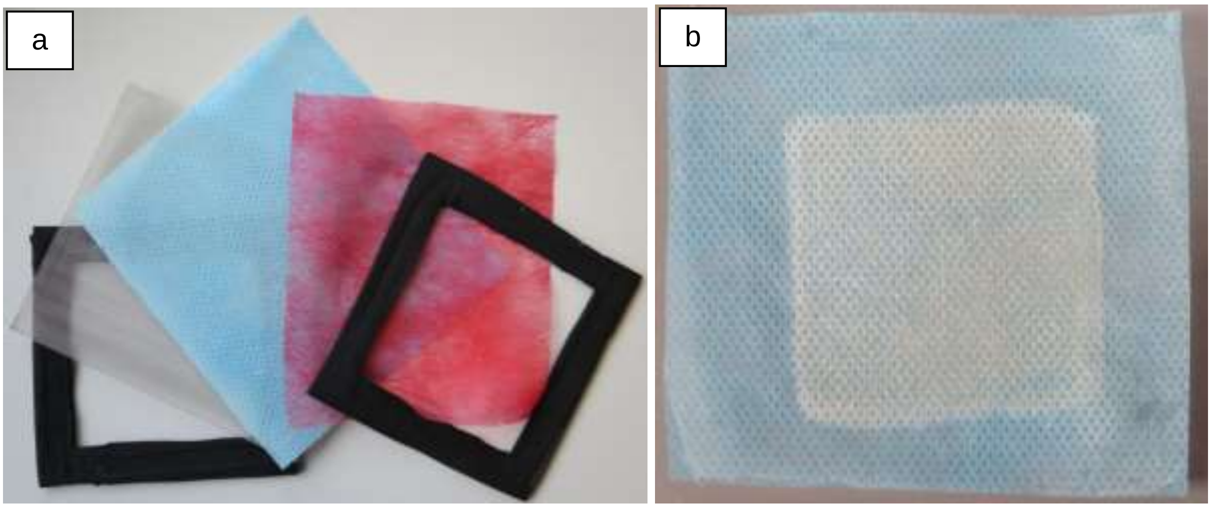

Fig. 2 a) Sample of a tested filter: Seals, stainless steel screen, nanofibrous web on polypropylene support (blue), protecting polypropylene textile (red), seals, b) Clogged nanofibrous textile by polystyrene particles after the experiment.

3.2. Visualization of a filtration process

During the filtration the camera records images of the filtration process and the images are saved to a PC. There the images were offline processed in the software ImageJ [5]. In the image behind a tested filter the evaluative areas are created to calculate local mean grey value. The local grey value is transform to the local mass concentration through a calibration constant. The mass concentration in front of a tested filter is known, it is the concentration of the prepared suspension: in this paper 100 µg per liter. More about the optical method for visualization and evaluation of a filtration process is in [6].

The flow rate was actively controlled and set on the constant value 0.3 liters per minute. Three experiments with the nanofibrous textile and with three different seeding particles were about 10 minutes long. The image analysis was carried out in the software ImageJ, where the global filtration efficiency was calculated.

3.3. Bubble point test

The bubble point test was carried out on the setup, which allows measuring the pressure drop and flow rate according to the norm: ASTM F316-A3. According to this test it is possible to observe the size of “flow pores”. The flow pore is defined as a space or tunnel, which connect both sides of the tested membrane.

The measurements were done on the device Macropulos 55. The size of investigated samples is 19.6 cm^2, the range of detectable pore sizes is 0.3 – 200 µm and the range of pressures is 0 - 0.6 MPa. The flow rate is increased step by step and the pressure drop was measured. The curve of flow rate versus pressure drop was measured on a dry sample at first, Fig. 3. The same curve was measured again on a wet sample (the sample is covered by a mineral oil). The first bubble point (the highest pressure during zero flow rate on the wet sample) signalizes the value of the pressure for the maximum pore size. The mean flow pore size was calculated from the pressure, where the wet curve crosses the half dry curve (all values of the flow rate for the dry curve are divided by two). It says in a fact that the half of the all pores in a filter is full of oil and the value expresses median of pore size. The dry curve is divided by 10 also and the intersection with the wet curve determines the pressure drop, from which 90 % pore size is calculated. It means that 90 % of all pores are smaller than this pore size. The maximum pore size is 15.45 µm, the mean pore size is 1.24 µm and the 90 % pore size is 2.46 µm. More information about bubble point test can be found in [7].

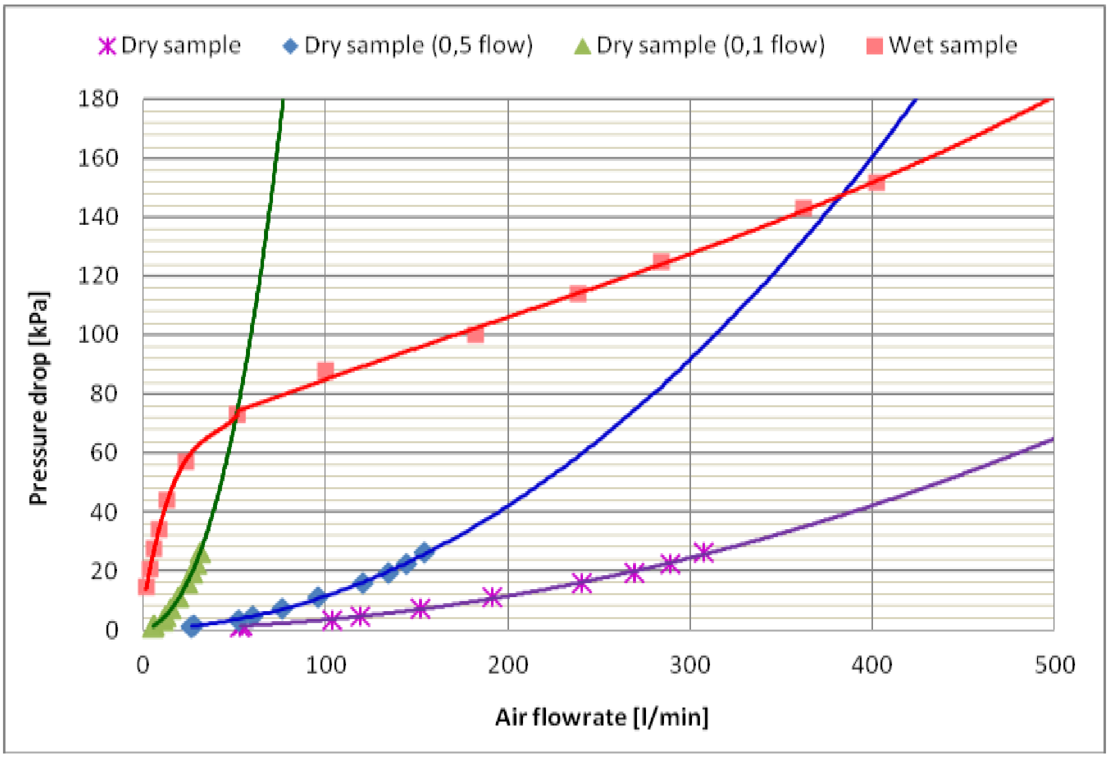

Fig. 3 Relation between airflow rate and pressure drop for dry and wet sample (mineral oil, 49.9 mN/m).

4. RESULTS

The pictures of the filtration process at the beginning of the experiments are shown in Fig. 4 – 6. We can see the inlet side of the filter, outlet side and the filter is placed in the middle. The inlet area is brighter due to the higher mass concentration, which is constant 100 µg per liter. The outlet area is brighter the more seeding particles pass through the filter. The 0.28 µm seeding particles pass through the nanofibrous textile very easily. The nanofibrous textile is very thick and the filtration efficiency is low, not higher than 10 %. The particles 0.42 µm are passing through the filter mainly by several holes. The filtration efficiency is around 50 %. The particles 0.69 µm are separated very well. The filtration efficiency is higher than 90 % and the outlet area is almost dark indicating low concentration of the seeding particles.

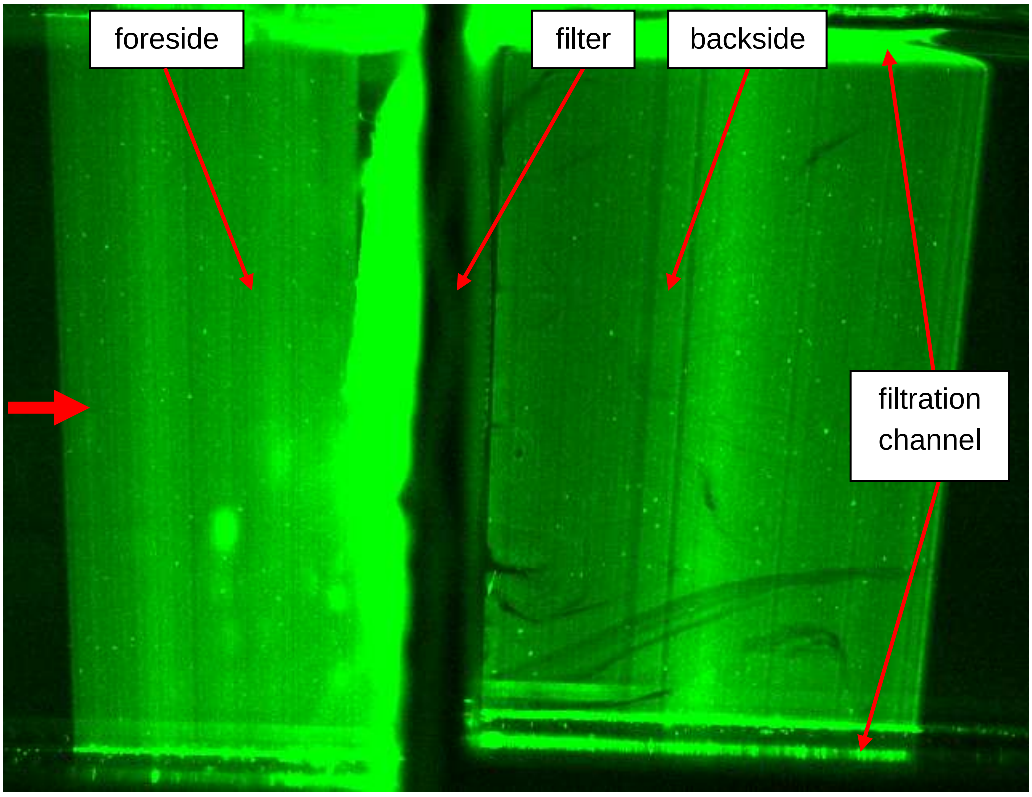

Fig. 4 Filtration process, water was seeded by 0.28 μm monodisperse polystyrene particles.

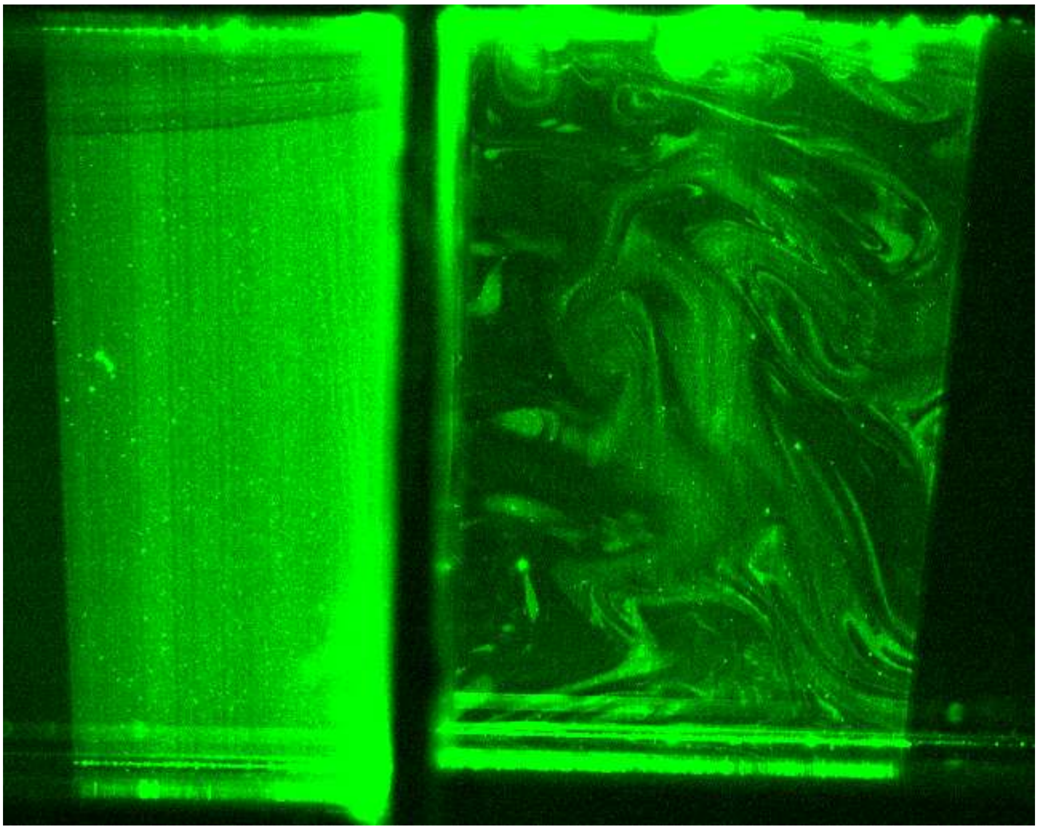

Fig. 5 Filtration process, water was seeded by 0.42 μm monodisperse polystyrene particles.

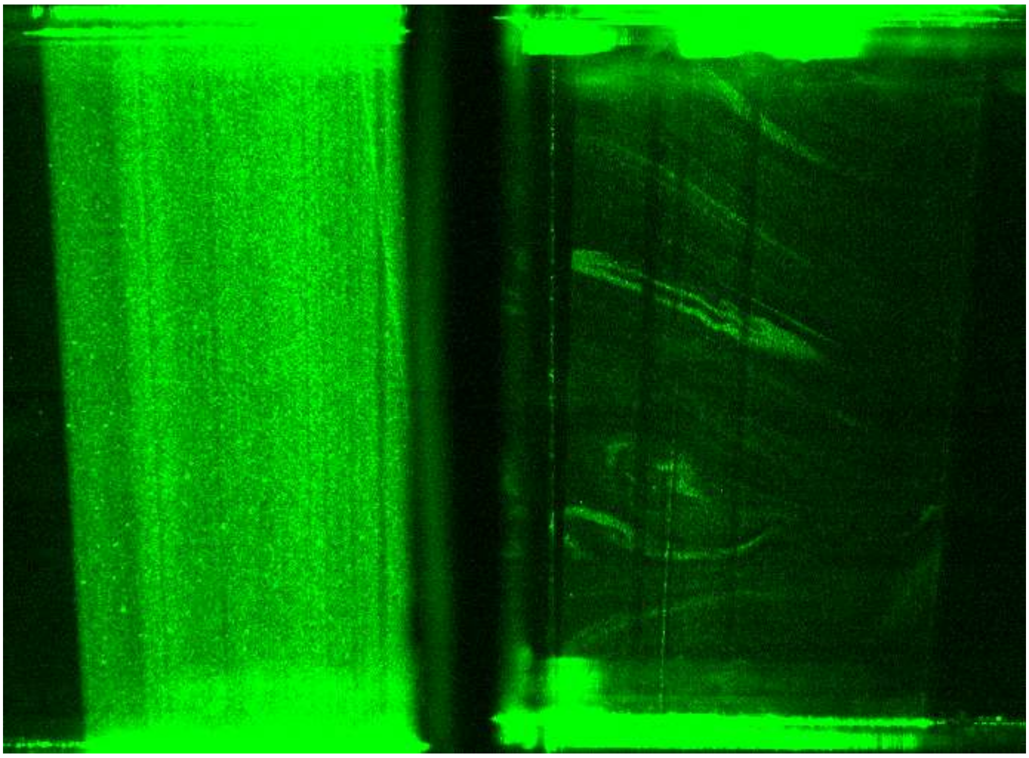

Fig. 6 Filtration process, water was seeded by 0.69 μm monodisperse polystyrene particles.

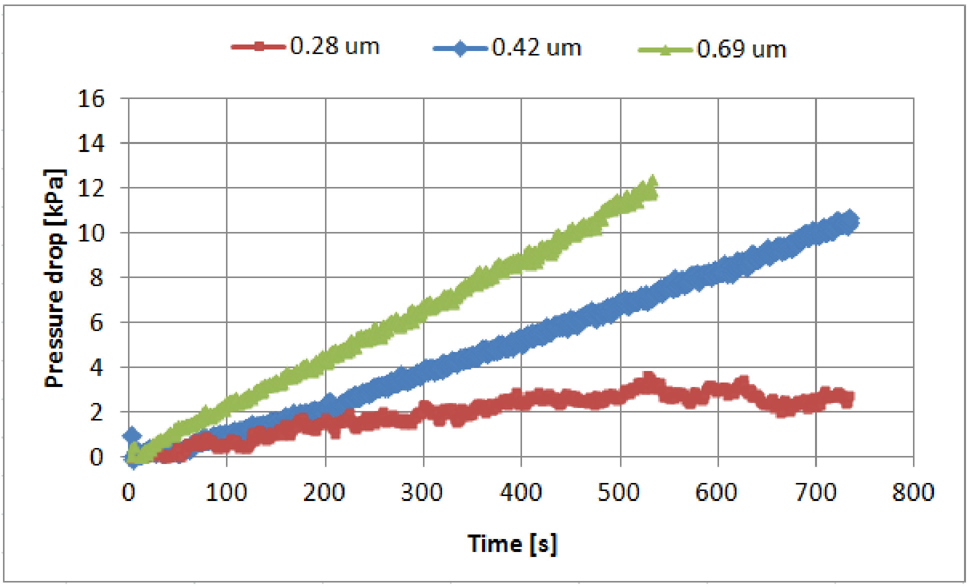

Fig. 7 Graph of pressure drop versus time for 3 types of the seeding particles.

The pressure drop versus time increases in case of all particle sizes, Fig. 7. The curves have linear character and the angular coefficient is the highest for the largest particles 0.69 μm and the smallest for the smallest particles 0.28 μm. This is caused by clogging of the fine nanofibrous web by bigger seeding particles and by creating of a filtration cake. The smallest particles are not trapped by the filter so easily and they mainly pass through. Very interesting are shapes of pressure courses. For bigger particle size pressure drop increases constantly but for small particle size (0,28 μm) the curve shape is more irregular. This shape shows that the particles captured inside the membrane are released again during the filtration process.

The maximum, mean and minimum pore size was compared to the filtration efficiencies. The mean pore size is 1.24 µm, approximately two times higher than seeding particles 0.69 µm. According to the visualization of a filtration process the filtration textile is able to catch most of the 0.69 µm large particles. This is probably caused by the effect, where at the same time two particles are forced to pass through a pore in a filter. The particles stick in the pore and cannot pass to the clean side of the filter. It is possible to say that flow pores inside the membrane are three-dimensional like an irregular tunnel. Thanks to this, the nanofibrous textile is able to separate even two times smaller particles than the mean pore size is. The same effect was found in other works [3].

5. CONCLUSION

The paper deals with a testing of a nanofibrous filter by submicron monodisperse particles on the basis of visualization of a filtration process. The method visualizates the whole filtration process by a laser sheet and a digital camera. By an image analysis it is possible to determine the local filtration efficiency versus time. On the images of the filtration process the local places with lower or higher filtration efficiency are seen. This was caused by non-uniform nanofibrous web, which includes places with lower or higher mass filling. The filtration efficiency for several sizes of seeding particles was compared to the maximum, mean and 90 % pore size measured by the bubble point test. It is obvious that the filtration textile is able to separate most of the particles, if their size is much smaller than the mean pore size. If the particle size is very small (less than the half value of the mean pore size), the filtration efficiency decreases very significantly. In the next experiments the nanofibrous filter will be tested by the particles larger than the mean pore size of the filter.

Another result of the paper is that the visualization method can serve as a verification method for the common used bubble point test. The bubble point test works on a different physical principle than the visualization of a filtration process. By the visualization of a filtration process it is possible to detect the weak places in a filtration material. The weak spots are in a fact the maximum pores.

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