DETERMINATION OF RESONANT FREQUENCIES OF NANOFIBROUS MEMBRANE BY HIGH-SPEED CAMERA

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Abstract

Nanofibrous layer performed as a membrane is capable of vibrating at low frequencies due to its small interfibrous spaces and planar arrangement brought into forced vibrations upon impact of sound waves of low frequency. The resonance of the nanofibrous elements allows acoustic energy to be converted into thermal energy. In this paper, a nanofibrous layer was produced by an electrospinning process from the water solution of polyvinyl alcohol (PVA). The PVA nanofibrous membranes of two different fiber diameters were made and the effect of membrane resonance has been studied. For a purpose of resonance and sound absorption properties of nanofibrous membrane, the two methods have been used. The optical method containing high-speed digital camera has been used for resonant frequencies of nanofibrous membrane determination. Two-microphone Impedance Measurement Tube Type 4206 was used to measure the sound absorption coefficient. Moreover, the nanofiber diameter of membrane has been evaluated.

Keywords: Nanofibrous membrane, resonance frequency, closed tube, fiber diameter, PVA

1. INTRODUCTION

Due to the possibility of resonating on its own resonant frequency the nanofibrous membrane is able to absorb critical lower sound frequencies. These unique properties come from the nature of nanofibrous layers, i.e. small fibrous diameter (respectively high specific surface area) and high porosity. This makes it possible to reach higher viscous loss inside the material and consequently to dissipate the acoustic energy. Nanofibrous elements and optimal rigidity of the membrane itself then allow an acoustic system to vibrate more efficiently [1, 2]. Resonant nanofibrous membranes of insignificant thickness are prepared from different polymers solutions in the form of electrospun nanofibers via electrospinning method.

The theoretical basis of sound absorption characteristics the paper deals with are studies performed by Sakagami et al. The study [3] focuses on a membrane-type sound absorber. The method used for predicting peak frequency and the peak value of the oblique-incident absorption coefficient of the membrane-type sound absorber is presented and satisfactorily explains the relationship between the absorption characteristics and the parameters. Resonant behavior of a micro perforated panel for various perforation ratios in comparison with a panel-/membrane-type absorber is presented in [4], considering back wall surface effect. In the papers [2,5] has been demonstrated that the nanofibrous layer has a resonant effect on sound absorption when the nanofibers are arranged with respect to the layer. The sound absorption peaks of longitudinally laid samples occur at frequencies lower than those of samples laid perpendicularly.

The effectiveness of a fiber-based sound absorbance material involves several parameters such as porosity, tortuosity, fiber diameter, surface density and thickness [6]. Kalinová has demonstrated that the resonance frequency of PVA nanofibrous membranes decreases with increasing surface density and average diameter of the nanofibers [2]. Comparing the sound absorption coefficient of electrospun silica fibers of different diameter to glass wool, Akasaka et al. found significant improvement in sound absorption of electrospun fibers over glass wool [7]. Rabbi et al. sandwiched PAN and PUR nanofibrous membrane between two nonwoven layers of PET and wool. All materials with electrospun membrane(s) were found to significantly increase its absorbance [8]. Asmatulu et al. tested the sound absorption of electrospun PVC mat of different thickness and with fiber diameters from a few hundred nanometers to a few microns. For fibers beyond 500 nm, the sound absorbance shift towards the lower frequency, but absorption peaks remain the same [1].

Vibration phenomena can be investigated by the noninvasive optical methods. The employment of the high-speed camera in the processes of vibration analysis has occurred over the past years in various application fields as well, including the analysis of human vocal fold vibrations, so-called videokymography [9]. Wang et al. combined the high-speed camera measurement with the finite element simulation method to determine the structural responses of materials on the full field vibration [10]. Ishizu et al. have studied the ossicular motion in the middle ear of pigs in response to acoustic stimulation using a high-speed camera [11]. In the study [12] sinusoidal fringe pattern on the measured drumhead is projected, dynamically deformed with the membrane vibration and grabbed by a high-speed camera. The paper [13] presents digital speckle correlation techniques, as an accurate analysis tool for 3D measurements of contours and displacements. Combination of laser method and high-speed camera is frequently used to obtain more detailed results. The paper [14] shows the experimental analysis of a semisolid and rectangular membrane by an out of plane interferometer setup that integrates a continuous wave with a fast camera. Nabavi describes the utilization of the particle image velocimetry technique to measure the velocity of the standing waves within an air-filled rigid-walled square channel subjected to acoustic standing waves [15]. This approach is related to the experimental setup used in this paper, but herein the interaction of the acoustic waves with nanofibrous membranes is studied. The recent study [16] shows how except for the lowest frequencies (first resonance peak), the resonant behavior of the membrane is affected by the resonance of tube when the effect of mass per unit area on resonance frequencies of the membrane placed in an open and closed tube is investigated. In general, the vibration of solid objects can be studied by laser methods. But the laser vibrometry, a sophisticated method, is limited - the precision of the measurement can be affected by material parameters such as surface roughness [17]. Moreover, the measurement of thin membranes which are translucent for the laser beam as in the case of the nanofibrous one, is another limitation. In this case, the laser methods are not so advantageous and high-speed camera analysis appears to be the best applicable method.

In accordance with the literature discussed above, the research is focusing on comparison between data obtained from the B&K impedance tube and high-speed digital camera analysis. The aim is to assess a relation between resonant behavior of PVA membrane of different fiber diameters and its sound absorption ability considering the effect of tube set-up.

2. EXPERIMENTAL PART

2.1. Materials

The water solution of polyvinyl alcohol PVA (Mw = 80.000 - 100.000 g/mol) was used for preparation of the solution for the experiment. Glyoxal and phosphoric acid were added as crosslinking agents. The content of glyoxal to PVA was 6 wt% and the content of phosphoric acid to PVA was 3 wt%. The concentration of the prepared PVA solution was 12.75 wt% and 14 wt%. The solution containing PVA, distilled water, glyoxal, and phosphoric acid was vigorously stirred at room temperature.

2.2. Production of nanofibrous membrane

For production of PVA nanofibrous membranes, roller electrospinning method (nanospider machine) was used [18]. In this method, there is a roller which is connected to high voltage supplier and top of the roller, where is a grounded counterelectrode. Taylor cones are then created on the roller surface, oriented toward the counterelectrode. Optimum process parameters such as roller speed, distance between the electrodes, voltage etc. were applied during the spinning process. The distance between the surface of the roll and the collector was 12 cm. Voltage of 50 kV, relative humidity of 34 %, and temperature of 19 °C were applied during the course of electrospinning. The final layer is crosslinked by hot air at the temperature of 140 °C for 5 min.

The average diameters of the produced nanofibrous membranes were changed using different speeds of support materials (**Table 1-2**).

Table 1 Parameters of the produced PVA nanofibrous membranes

PVA Solution Concentration [%]	Average Nanofiber Diameter [nm]	Area Density of the Membrane [g·m $^{-2}$]
12.75	**210**±113	5
14	**300**±118	5

Table 2 Average fiber diameter characteristics of the produced PVA nanofibrous membranes

PVA 12.75% (210 nm)					PVA 14% (300 nm)				
median [nm]	mean [nm]	st. deviation [nm]	min [nm]	max [nm]	median [nm]	mean [nm]	st. deviation [nm]	min [nm]	max [nm]
210	**213**	26	160	290	245	**296**	169	150	1170

2.3. Characterization

The fiber morphology and fiber diameter of the electrospun PVA fibers were determined using scanning electron microscopy (SEM). A small section of the fiber mat was placed on the SEM sample holder and sputter-coated with gold (Quorum Q150R Rotary-Pumped Sputter Coater). Carl Zeiss Ultra Plus Field Emission SEM using an accelerating voltage of 1.48 kV was employed to take the images (**Figure 1**).

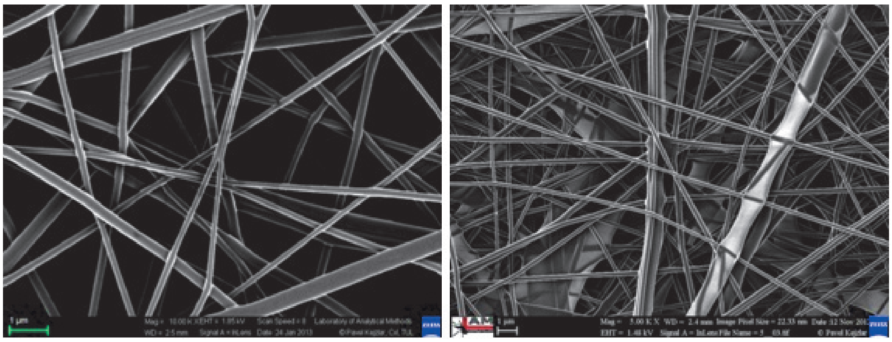

Figure 1 SEM images of morphology of 12.75% on the left and 14%PVA nanofibers on the right (1µm scale)

The average fiber diameter was calculated from the SEM images using image analysis software (NIS Elements BR 3.2). More than 100 fibers were counted from at least 4 SEM images which were taken from different places of a sample (**Figure 2**). Secondly, the nanofiber diameter influence on resonant frequency of membrane has been studied.

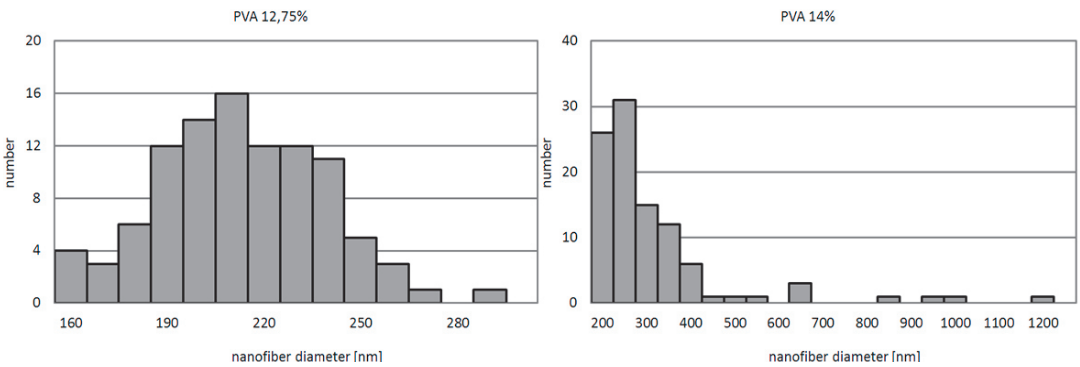

Figure 2 Diameter distribution of the PVA nanofibers prepared from 12.75% and 14% PVA solution

2.4. Methods for determination of resonant behavior

Sound absorbing ability in the frequency range of 50 Hz to 6.4 kHz was estimated in the Two-microphone Impedance Tube B&K (Type 4206). The amount of sound energy which is absorbed is described as the ratio of sound energy absorbed to the sound energy incident, and is termed the sound absorption coefficient (α).

The optical method was designed and verified by authors in the study [16]. The main components of the system that was used during the experiments are a digital camera (Olympus - System i-SPEED2), a LCD display panel of 8.4" and a transparent tube (see **Figure 3**).

During our study, 2000 frames/sec shooting was used at preset resolution of 800 x 600 pixels, each of which has a range of 200 shots per 0.1. A mark was drawn on the center of each sample in order to focus the lens of the camera. The tested sample was fixed in a position of 0.395 m from the sound source inside the tube of total length 0.62 m. A speaker at the end of the tube excited the incident plane sinusoidal sound wave. The membrane began to oscillate after the impact sound waves reached it. This movement was picked up by the high-speed digital camera and was displayed on the LCD screen. The position of membrane was chosen randomly at 0.637 of tube length to ensure that the membrane is not located exactly at node or antinode of tube. By placing the measured sample out of the characteristic resonant points, the basic resonant frequency of membrane should not be affected by the first resonant frequency of the tube at 139 Hz for the open tube and 277 Hz for the closed tube.

In order to determine the resonant frequency of the membrane, the 0-1000 Hz frequency range was studied by taking measurements at every 20 Hz to obtain a rough estimate of the resonant frequency. The deflection size of the nanofibrous membranes under the frequency range of 0-1000 Hz was measured using the following experimental set-ups: **open tube** (one end of the tube is open) and **closed tube** (the tube was closed with a rigid plate).

3. RESULTS

The **Figure 4** shows the comparison of resonance frequencies of the nanofibrous membranes of two fiber diameters in the open and closed tube arrangement. As may be seen from the graph, the deflection of

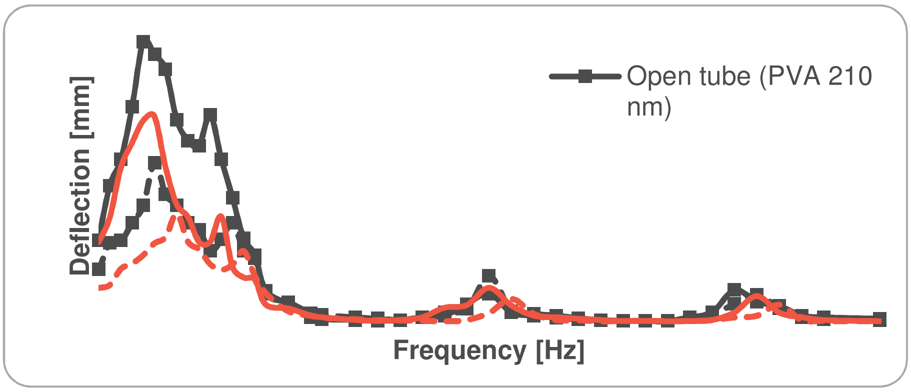

Figure 4 Deflection of the PVA membranes of two fiber diameters in the open and closed tube set-up

the membrane with smaller fiber diameter is higher than that with larger fiber diameter in the both arrangements (open and closed tube). It appears, the structure of smaller fiber diameter is more elastic.

The resonance curves of the membrane with smaller fiber diameter are then shifted towards the lower frequencies than that with larger fiber diameter in the both arrangements (open and closed tube).

The both nanofibrous membranes have been measured inside the two-microphone impedance tube in the distance of 20 mm from the back wall for the membrane vibrating ensure. From the **Figure 5** can be seen the deflection impact on the sound absorption. A higher sound absorption responds to the higher deflection of the membrane with smaller fiber diameter structure. It is evident the sound absorption depends on a motion degree of vibrating membrane during the sound impact as with a higher surface of the thinner fibers structure. On the other hand, the resonant frequency (peaks) displacement shows the opposite trend in both measurement methods. The resonant frequency of vibrating membrane doesn't respond to the sound absorption peak. The sound absorption maximal value of fibrous structure doesn't equal to the resonant frequency.

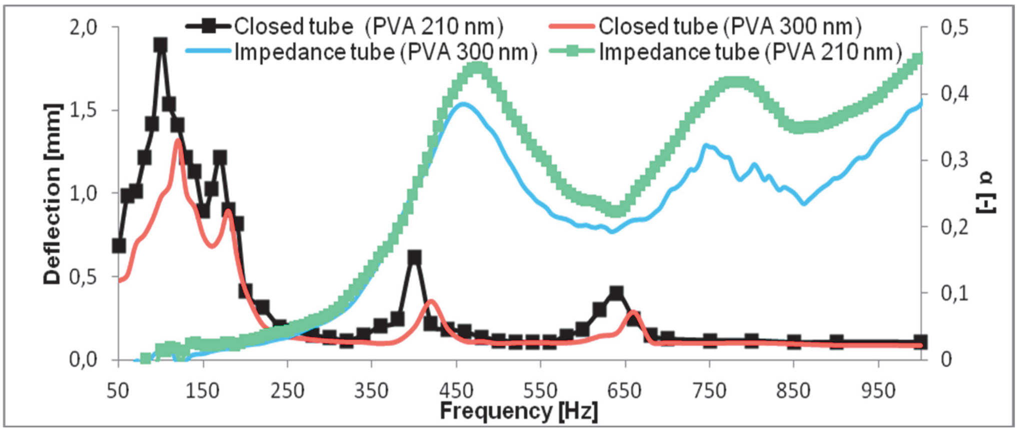

Figure 5 Effect of the closed method only. Comparison of deflection of the PVA membranes of two fiber diameters as dependence on sound frequency on the left and their sound absorption ability on the right

4. CONCLUSION

The PVA nanofibrous membranes of two different fiber diameters were made and the effect of membrane resonance has been studied. For a purpose of resonance and sound absorption properties of nanofibrous membrane, the two methods have been used. The optical method containing high-speed digital camera has been used for determination of resonant frequencies of nanofibrous membrane. The impedance tube was used to measure the sound absorption coefficient. A higher sound absorption responds to the higher deflection of the membrane with smaller fiber diameter structure. It was found the sound absorption depends on a motion size of the vibrating membrane during the sound impact - together with a higher surface of the thinner fibers structure. But, the resonant frequency doesn't respond to the sound absorption peak.

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REFERENCES

[1] ASMATULU, R., W. KHAN a M. B. YILDIRIM. Acoustical properties of electrospun nanofibers for aircraft interior noise reduction. In: *Proceedings of the ASME 2009 International Mechanical Engineering Congress & Exposition* [online]. 2009, pp. 223-227. ISBN 978-0-7918-4388-8. doi:10.1115/IMECE2009-12339

[2] KALINOVÁ, K. Nanofibrous resonant membrane for acoustic applications. *Journal of Nanomaterials* [online]. 2011, vol. 2011, pp. 1-6. ISSN 1687-4110, 1687-4129. doi:10.1155/2011/265720

[3] SAKAGAMI, Kimihiro, Masakazu KIYAMA, Masayuki MORIMOTO a Daiji TAKAHASHI. Sound absorption of a cavity-backed membrane: A step towards design method for membrane-type absorbers. *Applied Acoustics* [online]. 1996, vol. 49, no. 3, pp. 237-247. ISSN 0003682X. doi:10.1016/S0003-682X(96)00025-4

[4] SAKAGAMI, Kimihiro, Masayuki MORIMOTO a Motoki YAIRI. A note on the relationship between the sound absorption by microperforated panels and panel/membrane-type absorbers. *Applied Acoustics* [online]. 2009, vol. 70, no. 8, pp. 1131-1136. ISSN 0003-682X. doi:10.1016/j.apacoust.2009.03.003

[5] KALINOVA, K. Influence of nanofibrous membrane configuration on the sound absorption coefficient and resonant frequency. In: *Proceedings of the 6th AUTEX World Textile Conference*. 2006.

[6] SEDDEQ, Hoda S. Factors influencing acoustic performance of sound absorptive materials. *Australian Journal of Basic and Applied Sciences*. 2009, vol. 3, no. 4, pp. 4610-4617. ISSN 1991-8178.

[7] AKASAKA, Shuichi, Takahisa KATO, Shigeo ASAI a Hidetoshi MATSUMOTO. Sound absorption characteristics of silica nanofiber sheet. *Sound absorption characteristics of silica nanofiber sheet*. 2014, pp. 64-1.

[8] RABBI, A, K NASOURI, AM SHOUSHTARI a A HAJI. Fabrication of electrospun polyacrylonitrile and polyurethane nanofibers for sound absorption. In: *6TH TEXTEH INTERNATIONAL CONFERENCE* [online]. 2013, pp. 233-241. Available from: http://www.academia.edu/download/45476648/FABRICATION_OF_ELECTROSPUN_POLYACRYLONIT20160509-22247-3x9zsf.pdf

[9] INWALD, Elisabeth C., Michael DÖLLINGER, Maria SCHUSTER, Ulrich EYSHOLDT a Christopher BOHR. Multiparametric analysis of vocal fold vibrations in healthy and disordered voices in high-speed imaging. *Journal of Voice* [online]. 2011, vol. 25, no. 5, pp. 576-590. ISSN 08921997. doi:10.1016/j.jvoice.2010.04.004

[10] WANG, Weizhuo, John E. MOTTERSHEAD, Alexander IHLE, Thorsten SIEBERT a Hans REINHARD SCHUBACH. Finite element model updating from full-field vibration measurement using digital image correlation. *Journal of Sound and Vibration* [online]. 2011, vol. 330, no. 8, pp. 1599-1620. ISSN 0022460X. doi:10.1016/j.jsv.2010.10.036

[11] ISHIZU, K, T YASUI, M OHASHI, N MATSUMOTO a S KOMUNE. High-speed video analysis of acoustically oscillated guinea pig stapes. *The Journal of Laryngology & Otology* [online]. 2015, vol. 129, no. S2, pp. S33-S37. ISSN 0022-2151, 1748-5460. doi:10.1017/S0022215114002357

[12] ZHANG, Qican a Xianyu SU. High-speed optical measurement for the drumhead vibration. *Optics Express* [online]. 2005, vol. 13, no. 8, pp. 3110. ISSN 1094-4087. doi:10.1364/OPEX.13.003110

[13] SIEBERT, T, R WOOD a K SPLITTHOF. High speed image correlation for vibration analysis. *Journal of Physics: Conference Series* [online]. 2009, vol. 181, pp. 12064. ISSN 1742-6596. doi:10.1088/1742-6596/181/1/012064

[14] GUTIERREZ-HERNANDEZ, David Asael, Carlos PEREZ LOPEZ, Fernando MENDOZA SANTOYO, Alejandro TELLEZ-QUINONES a Daniel D AGUAYO. Optical characterization of a semisolid membrane by high speed interferometry. *Optoelectronics and Advanced Materials, Rapid Communications*. 2014, vol. 8, no. 5-6, pp. 382-386. ISSN 1842-6573.

[15] NABAVI, Majid, Kamran SIDDIQUI a Javad DARGAHI. Measurement of the acoustic velocity field of nonlinear standing waves using the synchronized PIV technique. *Experimental Thermal and Fluid Science* [online]. 2008, vol. 33, no. 1, pp. 123-131. ISSN 08941777. doi:10.1016/j.expthermflusci.2008.07.013

[16] KALINOVA, Klara, Merve K. OZTURK a Michal KOMAREK. Open and closed tube method for determination of resonance frequencies of nanofibrous membrane. *The Journal of The Textile Institute* [online]. 2016, vol. 107, no. 8, pp. 1068-1078. doi:10.1080/00405000.2015.1083353

[17] ROTHBERG, Steve J., Ben J. HALKON, Mario TIRABASSI a Chris PUSEY. Radial vibration measurements directly from rotors using laser vibrometry: The effects of surface roughness, instrument misalignments and pseudo-vibration. *Mechanical Systems and Signal Processing* [online]. 2012, vol. 33, pp. 109-131. ISSN 08883270. doi:10.1016/j.ymssp.2012.06.011

[18] JIRSAK, O., F. SANETRNIK, D. LUKAS, V. KOTEK, L. MARTINOVA a J. CHALOUPEK. Method of nanofibres production from a polymer solution using electrostatic spinning and a device for carrying out the method. *WO2005024101 (US2006290031)* [online]. 2009. Available from: http://www.google.com/patents/US7585437