THE EFFECT OF DEPOSITION TIME ON FILTRATION EFFICIENCY OF ELECTROSPUN NANOFIBRE WATER FILTERS

A.H. Nurfaizey[1], N.S.A. Roslan[1], M.I.M. Hafiz[1], M.A.M. Rosli[1], A.M. Saad[1] and N. Tucker[2]

[1]Faculty of Mechanical Engineering,
Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia.

[2]University of Lincoln, Brayford Pool, Lincoln,
LND 7TS, United Kingdom.

Corresponding Author's Email: [1]nurfaizey@utem.edu.my

Article History: Received 18 September 2018; Revised 10 July 2019;
Accepted 14 October 2020

ABSTRACT: Growing concern over turbidity of river waters due to high presence of solid suspensions has encouraged the development of new type of efficient water filters. In this study, a new type of water filter was developed by incorporating electrospun nanofibres. The relationship between the amount of incorporated nanofibres in term of deposition time and filtration efficiency was studied. Nylon 6 solution at 20 wt.% concentration was electrospun onto standard glass fibre filters. A high voltage of 14 kV was supplied at the spinneret and electrospinning distance was set at 10 cm. Suspended solid retention test was conducted on the glass fibre filters using a vacuum filtration system based on BS EN 872 standard. The morphology of the filters was studied using scanning electron microscopy and ImageJ software. From the results, the suspended solid retention capability increased linearly with nanofibre deposition time. Due to small size of the nanofibres, the addition of nanofibre layer has increased the total porosity of the filter. Findings from this study could open up further understanding in new generation of water filters.

KEYWORDS: *Electrospun Nanofibre; Electrospinning; Filtration; Suspended Solid; Deposition Time*

1.0 INTRODUCTION

Control of water pollution in Malaysia is facing a serious challenge due to rapid urbanization and industrial development. Certain synthetic materials used by the inhabitants may accumulate in the environment as they are non-biodegradable [1]. Moreover, residues or wastes disposed into the river streams could be incompletely degraded or removed during wastewater treatment [2]. For example, the increase of solid particles which persist in suspension in water also known as suspended solids would be harmful to aquatic life. Therefore, dissolved oxygen in water will be taken up and fewer living organisms could inhabit the polluted water [3]. To adapt to these challenges, filter manufacturers are continuously looking for ways to improve their filtration technologies

including the use of new class of materials. Submicron-sized fibres could be a potential candidate to replace fibrous materials used in conventional filters [4]. Electrospinning is a versatile and simple technique for producing polymeric fibres with submicron-range in diameter using electric charge [5-7]. Electrospun nanofibres can be formed into highly porous mesh with high specific surface area, good interconnectivity of pores and the potential to incorporate active materials [8]. These unique characteristics of electrospun nanofibres are desirable for many applications including filtration. Previous studies have reported the potential applications of electrospun nanofibre as high-performance air filters, protective clothing, sensors, wound dressing, sunscreens [9] advanced composites, photovoltaic cells, scaffolds in tissue engineering [10] and more recently as membranes in affinity separation [11].

Shin [12] reported the potential of electrospun nanofibre water filtration membrane made from recycled polystyrene. Standard glass fibre filters were used with addition of nanofibres. The result showed that separation efficiency was improved with the inclusion of nanofibres from 61% to 91%. Zander et al. [13] used recycled PET nanofibres for water filtration applications. It was reported that the pore size was found to correlate with fibre diameter and the smallest fibres showed the best filtration capabilities. Another recent study from Noorpoor et al. [14] suggested that the total filtration efficiency is proportional to the thickness of the filter media.

In this study, the objective is to investigate the effect of nylon 6 electrospun nanofibres inclusion on suspended solid filtration efficiency. Nylon 6 is thought suitable for filtration applications because of its good resistance to numerous commercial solvents [15]. The knowledge gained from this study is important for developing new generation of efficient water filtration media.

2.0 METHODOLOGY

2.1 Electrospinning Process

Electrospinning process was carried out using a laboratory scale electrospinning machine (Model ES1a, Electrospinz, NZ) as shown in Figure 1. Polymer solution was prepared by dissolving nylon 6 pellets (Sigma-Aldrich 181110) in formic acid (Merck 1002642500) to a final concentration of 20 wt.%. An applied voltage of 14 kV was kept constant throughout the electrospinning process and the electrospinning distance was set at 10 cm.

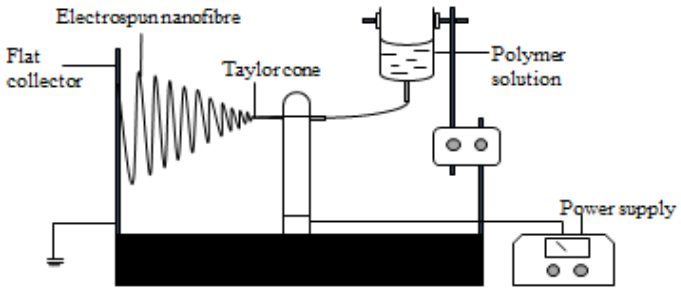

Figure 1: A typical electrospinning apparatus

Standard glass fibre filters with pore size of 1.2 μm and diameter of 47 mm (Merck & Co., USA) were used as the substrate materials. Addition of nanofibres was done by directly electrospinning nylon 6 fibres onto the standard filters. Electrospinning was performed at different deposition times of 0 (standard filter), 20, 40, 60, 80, 100 and 120 seconds to study the effect of deposition time on filtration efficiency. Triplicate samples were prepared for each case and labelled as sample F1 (standard filter), F2, F3, F4, F5, F6 and F7, respectively.

2.2 Retention Test

The efficiency of the filter samples was tested based on the amount of captured suspended solid (BS EN 872 standard) using suspended solid vacuum filtration system (Hach Co., USA) (Figure 2). In each test, a filter sample was put onto the filtering flask and 100 ml of water sample was poured through the filter with the assistance of a pump. Filter samples were then dried in an oven at 102-105 °C (217–221 °F) for about 2 hours. Filter samples were weighed before and after filtration using four figures balance Model AG204 (Mettler Toledo, Switzerland). Water sample was taken from a water treatment plant at UTeM Main Campus.

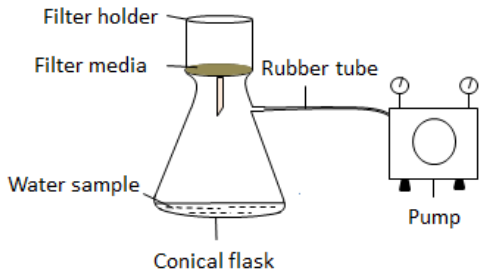

Figure 2: A schematic diagram of a vacuum filtration test system

2.3 Characterization of Filter Media

Physical characterization of the filters was carried out using scanning electron microscope (SEM) Model JSM-6010PLUS/LV (JEOL Ltd., Japan). Samples were coated with a conductive platinum layer using JEC-3000FC Auto Fine Coater (JEOL Ltd., Japan) for 300 seconds. Using ImageJ v1.5 software, at least 50 measurements of fibre diameter were made from each SEM micrograph to determine the average fibre diameter of the samples.

Surface porosity of the filters were estimated using image analysis method from SEM micrographs using ImageJ software whilst total porosity of the filters were determined using gravimetric method based on the following equation as shown in [15-16]:

$$E(\%) = \frac{w_w - w_d}{\rho_e AL} \times 100 \quad (1)$$

where W_w and W_d are the weight of the wet and dry filter respectively, ρ_e is the density of ethanol at room temperature (0.789 g/cm^3), A is the effective area of the filter, and L is the filter thickness. W_w was obtained by immersing the samples in ethanol for 1 hour. Then, excess ethanol was removed from the surface using tissue paper. The samples were weighed using four-figure balance to measure W_w. After weighing, the samples were dried in an oven at 70 ± 0.3 °C overnight. After that the samples were weighed again to determine the value of W_d. The average weight of wet and dry samples were calculated, and the porosity was determined according to Equation (1) [17]. The thickness of the samples was obtained using a micrometre (Mitutoyo Co., Japan).

3.0 RESULTS AND DISCUSSION

The values of total suspended solid (TSS) retention were calculated for each sample (Table 1). TSS value when using the standard filter (F1–control sample) was 4 mg/L. A steady increase of TSS value was observed (TSS 7 to 23) when using Sample F2, F3, F4, F5, F6 and F7 filters (Figure 3). The results suggest that the increase of electrospinning deposition time improved the capability of the filters in capturing suspended solids. This result is in agreement with a study by Alharbi et al. [18].

Table 1: Total suspended solid (TSS) retentions using BS EN 872

Sample	F1	F2	F3	F4	F5	F6	F7
Deposition time (s)	0	20	40	60	80	100	120
Weight (before) (mg)	99.0	99.3	100.3	101.1	101.4	101.8	102.0
Weight (after) (mg)	99.4	100.0	101.3	102.5	103.2	103.8	104.3
TSS (mg/L)	4.0	7.0	10.0	14.0	18.0	20.0	23.0

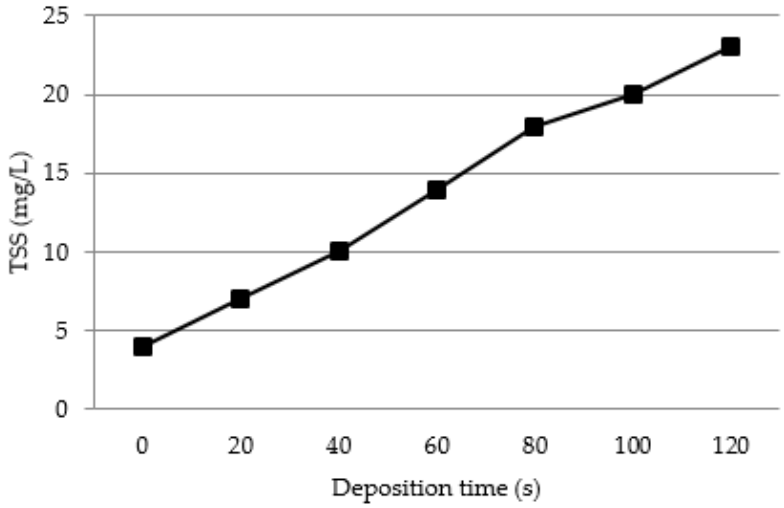

Figure 3: TSS retentions of the samples at different deposition times: 0, 20, 40, 60, 80, 100 and 120 seconds

The average fibre diameter of the nylon 6 nanofibres was 173.63 nm whilst the diameter of the substrate fibres was up to several micrometres (Figure 4). In filtration applications, smaller fibre is more desirable in order to provide greater surface area, higher porosity, and increased product efficiency [13].

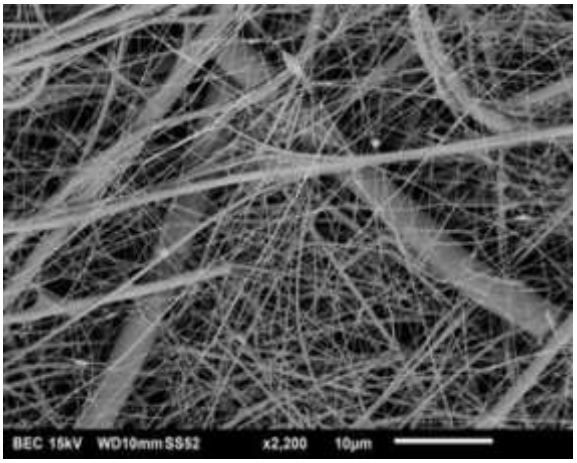

Figure 4: SEM micrograph (×2200 magnification) of the filter samples showing fine nylon 6 electrospun nanofibres overlaying glass microfibres of the substrate material

Physical differences of the filter samples are shown in Figure 5. As can be seen in Figure 5 (a), there were no nylon 6 nanofibres in the control sample. However, for deposition time t = 20 s, a small amount of nanofibres can be seen bridging between the substrate's microfibres. In general, more nanofibres can be seen as deposition time increased. This explains the results obtained in Figure 3, where the increase of deposition time will increase the amount of porous nanofibre network, thus increasing the capability of the filter to capture suspended solids.

The thickness, average of W_w and W_d and total porosities of the filters are shown in Table 2. The thickness of the control filter with no nanofibres was 0.150 mm. However, filter thickness increased from 0.170 to 0.240 mm as the deposition time increased. The overall results show that total porosity of the samples increased as electrospinning deposition time increased. The results suggest that as more porous network created between the fibres, the capability of the filter to absorb liquid increases thus increasing the total porosities. However, there was a slight decrease from F1 to F2. This is thought due to some abnormalities during the measurements of W_w and W_d.

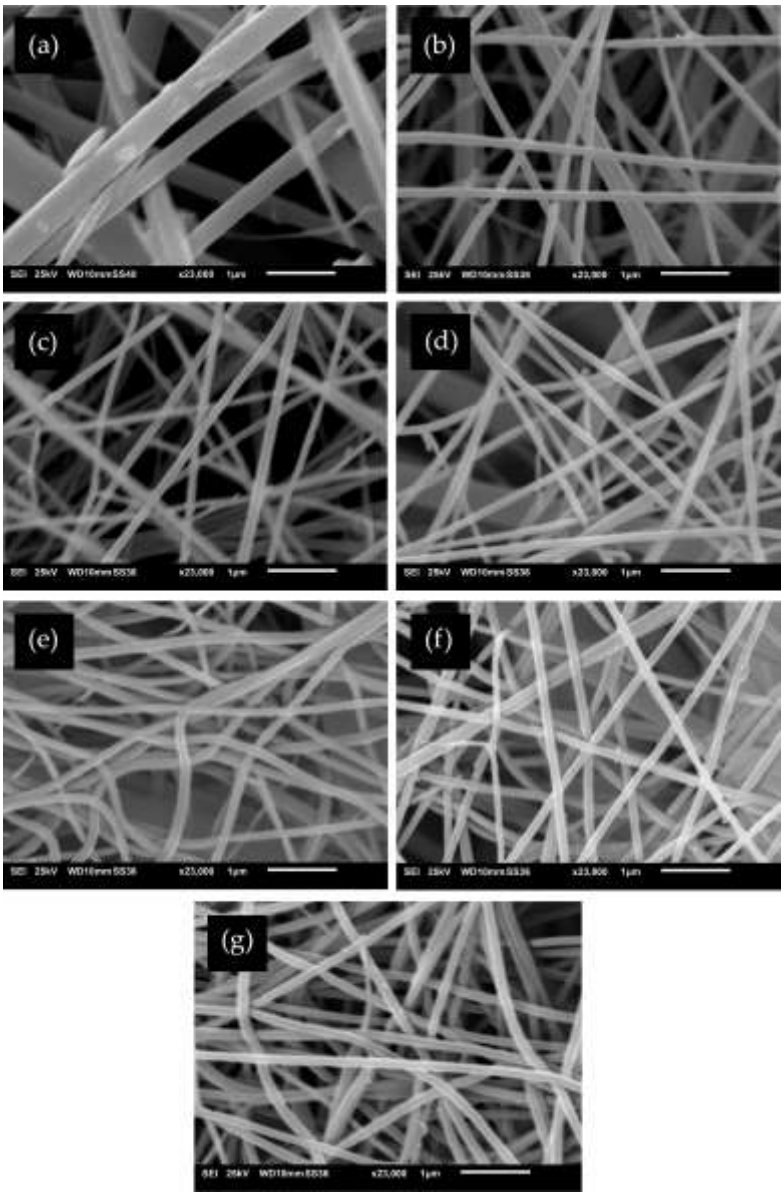

Figure 5: SEM micrographs showing the physical differences of the filter samples (×23000) at deposition times of (a) 0 sec, (b) 20 sec, (c) 40 sec, (d) 60 sec, (e) 80 sec, (f) 100 sec and (g) 120 sec

Table 2: Thickness, W_w and W_d and total porosity of the filter samples

Sample	Thickness (mm)	Ww(g)	Wd(g)	Total porosity (%)
F1	0.150	0.2166	0.0880	62.63
F2	0.170	0.2257	0.0983	54.74
F3	0.185	0.2842	0.1007	78.87
F4	0.188	0.3101	0.0997	83.07
F5	0.207	0.3358	0.1009	82.93
F6	0.223	0.3667	0.1024	86.57
F7	0.240	0.4042	0.1284	83.94

The relationship between electrospinning deposition time, total porosity, and surface porosity is presented in Figure 6. Surface porosity is defined as the ratio of void to the total surface area of the sample while total porosity is defined as volume of void to the total volume of sample [19]. As expected, when deposition time increased, the surface porosity decreased. This was because as more nanofibres were deposited onto the filter, there were more interconnectivities between the fibres causing the voids to decrease. However as mentioned earlier, the total porosity of the filters was increased with deposition time (Figure 6). Even though more deposited fibres reduced the surface porosity, it is thought that the overall 3D network formed by the nanofibres has created a very porous medium. The results obtained is in agreement with the study conducted by Correia et al. [16].

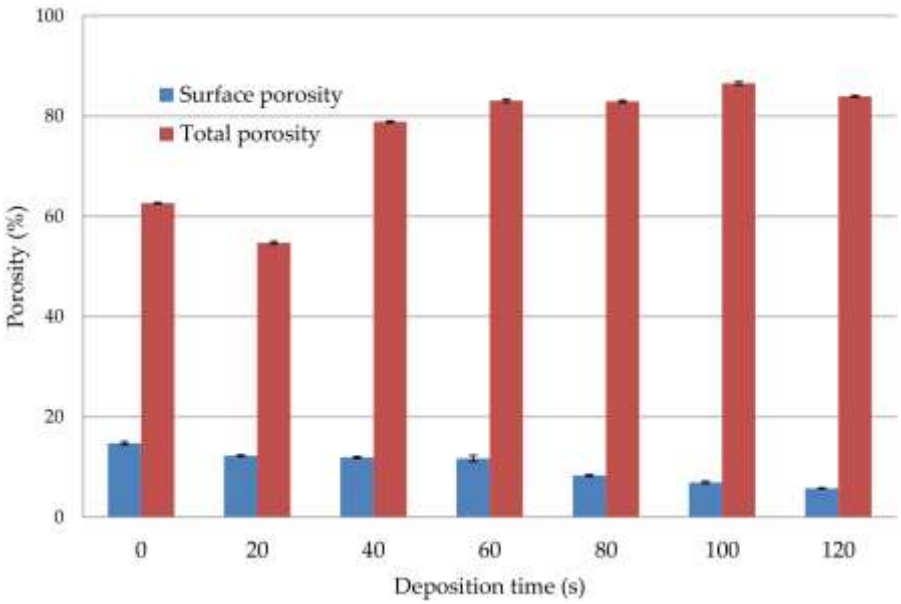

Figure 6: Relationship between deposition time, surface porosity and volume porosity of the filter samples

The results gained from this study could provide useful information for the development of new type of water filtration system. Further works on characterizing and evaluating the performance of the proposed filters based on liquid permeability and pore size are ongoing and the results will be reported later.

4.0 CONCLUSION

In this study, electrospinning technique was used to incorporate nylon 6 electrospun nanofibres onto standard filters. The effect of electrospinning deposition time on filtration efficiency of the filters was studied. From the results, the amount of trapped suspended solid was increased with increasing deposition time. The results suggest that the greater amount of nanofibres incorporated onto the filters will improve the capability of the filters. From SEM micrograph, more nanofibres can be seen as deposition time increased. Surface porosity decreased as deposition time increased. However, total porosity of the filters increased with deposition time. The results from this study would contribute to the existing literature in nanofiltration technology.

ACKNOWLEDGEMENTS

Special thanks to the Ministry of Higher Education Malaysia and Universiti Teknikal Malaysia Melaka for funding this study through PJP/2015/FKM(2A)/S01397. Thanks also to the Advanced Materials Characterization Laboratory (AMCHAL) group and Centre for Advanced Research on Energy (CARe) for their supports.

REFERENCES

[1] N. D. Gopal, P. Phebe, E. S. Kumar, and B. K. K. Vani, "Impact of plastic leading environmental pollution," *Journal of Chemical Pharmaceutical Sciences*, vol. 3, pp. 96–99, 2014.

[2] S. Barnabé, I. Beauchesne, D. G. Cooper, and J. A. Nicell, "Plasticizers and their degradation products in the process streams of a large urban physicochemical sewage treatment plant," *Water Research*, vol. 42, no. 1–2, pp. 153–162, 2008.

[3] A. D. Gosomji and M. A. Okooboh, "Determination of the Concentration of Dissolved Oxygen in Water Samples from Pankshin Town to Monitor Water Pollution," *Chemistry and Materials Research*, vol. 3, no. 3, pp. 13–17, 2013.

[4] N. S. A. Roslan, A. H. Nurfaizey, M. H. M. Isa, N. Muhammad, M. R. Mansor, and N. A. Munajat, "Nylon electrospun nanofibre water filtration media for wastewater treatment," *Material Research Express*, vol. 5, no. 10, pp. 1-11, 2018.

[5] A. H. Nurfaizey, F. C. Long, M. A. M. Daud, M. R. Mansor, N. Tucker, N. Muhammad, "An investigation of using grey scale image analysis for predicting the amount of deposited electrospun nanofibres," *Journal of Mechanical Engineering and Sciences,* vol. 13, no. 1, pp. 4679-4692, 2019.

[6] Z. M. Huang, Y. Z. Zhang, M. Kotaki, and S. Ramakrishna, "A review on polymer nanofibers by electrospinning and their applications in nanocomposites," *Composites Sciences and Technology*, vol. 63, no. 15, pp. 2223–2253, 2003.

[7] T. Subbiah, G. S. Bhat, R. W. Tock, S. Parameswaran, and S. S. Ramkumar, "Electrospinning of nanofibers," *Journal of Applied Polymer Science*, vol. 96, no. 2, pp. 557–569, 2005.

[8] S. Ramakrishna, K. Fujihara, W. E. Teo, T. Yong, Z. Ma, and R. Ramaseshan, "Electrospun nanofibers: Solving global issues," *Materials Today*, vol. 9, no. 3, pp. 40–50, 2006.

[9] S. Sundarrajan, K. L. Tan, S. H. Lim, and S. Ramakrishna, "Electrospun nanofibers for air filtration applications," *Procedia Engineering*, vol. 75, pp. 159–163, 2014.

[10] R. Gopal, S. Kaur, Z. Ma, C. Chan, S. Ramakrishna, and T. Matsuura, "Electrospun nanofibrous filtration membrane," *Journal of Membrane Science*, vol. 281, no. 1–2, pp. 581–586, 2006.

[11] Z. Ma, M. Kotaki, and S. Ramakrishna, "Electrospun cellulose nanofiber as affinity membrane," *Journal of Membrane Science*, vol. 265, no. 1–2, pp. 115–123, 2005.

[12] C. Shin, "Filtration application from recycled expanded polystyrene," *Journal of Colloid and Interface Science*, vol. 302, no. 1, pp. 267–271, 2006.

[13] N. E. Zander, M. Gillan, and D. Sweetser, "Recycled PET nanofibers for water filtration applications," *Materials*, vol. 9, no. 4, pp. 1–10, 2016.

[14] A. R. Noorpoor, A. Sadighzadeh, and A. Anvari, "Effect of nylon-6 concentration on morphology and efficiency of nanofibrous media," *International Journal of Environmental Research*, vol. 8, no. 2, pp. 421–426, 2014.

[15] J. Deitzel, J. Kleinmeyer, D. Harris, and N. B. Tan, "The effect of processing variables on the morphology of electrospun nanofibers and textiles," *Polymer*, vol. 42, no. 1, pp. 261–272, 2001.

[16] T. R. Correia, B. P. Antunes, P. H. Castilho, J. C. Nunes, M. T. P. de Amorim, I. C. Escobar, J. A. Queiroz, I. J. Correia, and A. M. Morão, "A bi-layer electrospun nanofiber membrane for plasmid DNA recovery from fermentation broths," *Separation and Purification Technology*, vol. 112, pp. 20–25, 2013.

[17] M. Obaid, Z. K. Ghouri, O. A. Fadali, K. A. Khalil, A. A. Almajid, and N. A. M. Barakat, "Amorphous SiO2 NP-Incorporated Poly(vinylidene fluoride) Electrospun Nanofiber Membrane for High Flux Forward Osmosis Desalination," *ACS Applied Materials and Interfaces*, vol. 8, no. 7, pp. 4561–4574, 2016.

[18] A. R. Alharbi, I. M. Alarifi, W. S. Khan, and R. Asamatulu "Highly hydrophilic electrospun polyacrylonitrile/polyvinypyrro-lidone nanofibers incorporated with gentamicin as filter medium for dam water and wastewater treatment," *Journal of Membrane and Separation Technology*, vol. 5, no. 2, pp. 38–56, 2016.

[19] V. Gitis and G. Rothenberg, *Ceramic membranes: new opportunities and practical applications*. Weinheim: Wiley, 2016.