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Remediation of pollutions with oily products using polymer-based fibers as sorbents

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Abstract. Fibrous polymeric materials gained particular interest, owing to their significant contact surface area and tunable porous morphology. A fibrous–porous sorbent based on electrospun poly(vinylidene fluoride-co-hexafluoropropylene) was fabricated and investigated for oil sorption applications. The copolymer was electrospun under optimized operating conditions, aiming to obtain a porous, hydrophobic nonwoven composed of submicronic fibers. The material was characterized by FT-IR, EDAX, SEM, and mechanical testing. Its sorption performance was assessed for the uptake of the motor oil (0.843 g/mL, 67 cP), yielding a sorption capacity of 9.3 g/g, and oil recovery from the spent sorbent was demonstrated by centrifugation. Additionally, a comparative analysis was performed between the electrospun nonwovens (submicronic fibers) and conventional textile nonwovens (microfibers). Thus, it was pointed out that the influence of fiber diameter and inter-fiber spacing affects the sorption capacity and oil recovery efficiency by centrifugation.
Keywords: electrospinning method, polymers, oil sorption, process optimization.

Remedierea poluărilor cu produse uleioase utilizând fibre pe bază de polimeri ca şi adsorbanţi

Rezumat. Materialele polimerice fibroase prezintă un interes deosebit datorită suprafeţei lor de contact relevante şi a morfologiei poroase reglabile. A fost investigat un sorbent fibros-poros pe bază de poli(fluorură de viniliden-co-hexafluoropropilenă) obţinut prin metoda electrofilării, fiind destinat pentru aplicaţii de sorbţie a produselor uleioase. Copolimerul a fost electrofilat în condiţii de operare optimizate având drept scop obţinerea unui un material neţesut poros, hidrofob, compus din fibre submicronice. Materialul a fost caracterizat prin FT-IR, EDAX, SEM şi teste mecanice. Performanţa de sorbţie a fost evaluată pentru colectarea uleiului de motor (0,843 g/mL, 67 cP), rezultând o capacitate de sorbţie de 9,3 g/g, iar recuperarea uleiului din sorbentul epuizat a fost posibilă prin centrifugare. În plus, a fost efectuată o analiză comparativă înre materialele neţesute electrofilate (fibre submicronice) şi materialele neţesute textile convenţionale. Astfel, s-a evidenţiat că influenţa diametrului fibrelor şi a spaţierii dintre fibre afectează capacitatea de sorbţie şi eficienţa de recuperare a uleiului prin tehnici de centrifugare.
Cuvinte-cheie: metoda electrofilării, polimeri, sorbţia produselor uleioase, optimizarea proceselor.

1. Introduction

Oil spills occur frequently and represent accidental environmental pollution with hydrocarbons and oily products arising from the processing, transportation, and storage of oils [1, 2, 3, 4]. From the existing methods for combating oil spill pollution, one practical approach uses sorbent materials. These are typically classified as inorganic, natural organic, synthetic, and composite sorbents [2, 3, 4]. All of these material types have their own advantages and limitations. Over the last two decades, polymer-based fibrous sorbents (including composites) have attracted considerable interest [1]. This behaviour may be attributed to the fibrous morphology, which increases the contact surface area between the adsorbent and the oily pollutant and facilitates oil uptake within the *inter-fiber spaces* or *voids* (V_{IF}), which act as effective *pores* where the liquid phase is entrapped.

Previous work [2] identified three types of liquid fractions that can be retained by fibrous–porous scaffolds: (1) the *drainable liquid* fraction, (2) the *capillary-retained* liquid fraction, and (3) the *adherent liquid film*. By analogy, this classification allows the adoption of capillary pore ranges commonly used in hydrology, where liquid flow through soil capillaries is extensively studied [5].

Consequently, the drainable liquid fraction in fibrous–porous scaffolds can be associated with the excess liquid carried by cohesive forces as well as with the liquid fraction entrapped in *supra-capillary pores* ranging from 500 to 1200 μm. Within these *supra-capillary pores*, liquid transport follows hydrodynamic laws. For example, when a saturated fibrous–porous sorbent is removed from the liquid phase (*e.g.*, oil bath), the drainable liquid fraction is rapidly released from these liquid-filled pores under gravitational forces, typically within the first moments (0.5–2 min). The capillary-retained liquid fraction is thereby trapped within *capillary pores* with sizes ranging from 0.2 to 500 μm. In these pores, the liquid may circulate according to the capillary phenomena or remain retained by capillary forces. The capillary-retained liquid fraction can be removed from a spent porous sorbent *via* squeezing or centrifugation. Lastly, the adherent liquid fraction is attached to the solid surface by adsorptive forces or retained by very strong capillary forces within *sub-capillary pores* with sizes smaller than 0.2 μm. In such small pores (< 0.2 μm), liquids cannot circulate *via* capillary phenomena. Consequently, the adherent liquid fraction cannot be removed from the spent sorbent by centrifugation and can only be eliminated by evaporation at elevated temperatures.

The previous study [4] demonstrated the possibility of hydrophobization of polyester (PESt) microfibers assembled in the form of nonwoven materials, originating from the

textile industry. In this regard, polyester microfibers with diameters in the range ∅ = 10–50 μm were coated with a thin polysiloxane (PSi) layer (~5 μm). After hydrophobization, these fibrous nonwoven materials (PESt@PSi) exhibited good performance in removing oily layers (1–5 mm) from the water surface [4]. For the removal of thinner oil slicks (< 1 mm) and even oily shines or iridescences, polymer-based fibrous membranes/nonwovens produced by electrospinning can be employed [2, 3]. This method can produce ultrathin polymeric fibers with diameters of several microns, or even submicronic fibers (*i.e.*, of hundreds of nanometers in diameter). Hence, the electrospinning operates at the micro- and nanoscale to manufacture the advanced nonwovens based on ultrathin fibers [6, 7].

The electrospinning process is influenced by a series of experimental factors: (1) operating parameters ensuring the electrospinning (*i.e.*, applied high voltage, needle-collector distance, and feed flow-rate); (2) factors related to the polymeric solution (*i.e.*, type of solvent and polymer, concentration and molecular weight of the polymer, solution viscosity, and surface tension); and (3) ambient factors (temperature and air humidity). To produce the micro/nano fibers through electrospinning, the polymeric solution should possess a minimum electrical conductivity. This depends on the type of solvent, type of polymer, polymer concentration, and temperature. The polymeric solutions with zero electrical conductivity cannot undergo electrospinning. Likewise, the possibility of electrospinning depends on the viscosity of the solution, which is proportional to the polymer concentration. For instance, if the viscosity of the polymeric (dope) solution is too high (e.g., polymer concentration > 25 wt.%), the jet may not be expelled from the needle tip. In turn, a quite diluted polymer solution (< 10 wt.%) may interrupt the process by discontinuing the jet, and no fibers will be formed. As regards the operating parameters, typically, a high *applied voltage* (U, kV) yields ultrathin fibers with decreased diameters, owing to great electrostatic repulsion forces on the liquid jet. However, extremely high voltage can unexpectedly induce fibers with larger diameters, or sometimes, even unfavorable beads may appear alongside the fiber surfaces.

Other key operating parameters imply the *needle-collector distance* (Λ, cm) and the *feed flow rate* (Q, mL/h). The needle-to-collector distance influences electric field strength, jet stretching, solvent evaporation, and final fiber morphology. Usual distances in practice are 10–20 cm [1, 2, 3, 7], which are sufficient for balancing electrostatic forces for Taylor cone formation and stable jet travel. Shorter distances (<10 cm) contribute to strong fields but insufficient evaporation time, and this yields to wet, beaded, or fused fibers. Longer distances (>20 cm) weaken the electrostatic field, which has as result the less uniform fibers as the jet loses drawing forces. The feed flow rate of the dope solution, containing the solvent, polymer, and optionally additives, affects the mass transfer and the velocity

of the jet [8]. The uniformity of electrospun fibers, as well as their diameters, can be tuned by adjusting the experimental factors and operating parameters [9]. Hence, each experimental factor or operating parameter is important, and it should be considered in the design and preparation of the electrospun materials with adequate morphology for a given application.

2. Results and discussion

The electrospinning setup used for experimentation is illustrated in Figure 1. As shown in this figure, electrospinning relies on an electrically charged liquid jet (polymer-based solution) ejected from a syringe with a metallic needle under a high voltage (kV scale).

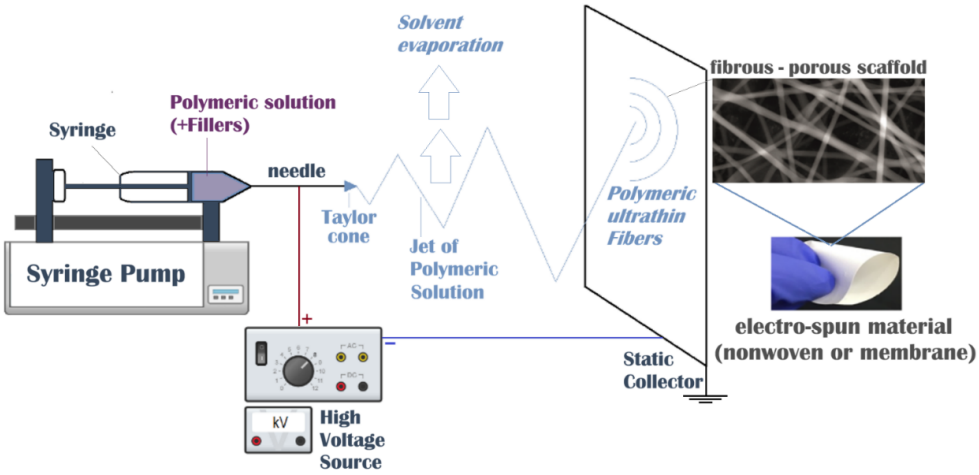

Figure 1. Scheme of the electrospinning setup used in the experiments

According to Figure 1, at the tip of the needle (subjected to high voltage), the attached droplet is elongated by the induced charge, forming the Taylor cone [2] from which the fluid jet is expelled. As this jet travels the needle-to-collector distance, solvent evaporation occurs, yielding ultrathin polymeric fibers deposited onto the surface of the grounded collector (Figure 1). In the first stage, the objective was to optimize the operating parameters, namely, the applied voltage (U, kV), needle-to-collector distance (Λ, cm), and feed flow rate (Q, mL/h), as reported in our previous work [2]. To this end, a design of experiments (DoE) approach based on a full factorial design (FFD) was employed in the frame of the response surface methodology (RSM). This strategy enabled the development of two chemometric models correlating the sorption performance (S, g/g) of the electrospun material with the operating parameters (U, Λ, and Q) used during its fabrication. Thus, the polysulfone-based electrospun materials prepared by Cojocaru *et*

al. [2] under the various conditions, *i.e.*, U (20–25 kV), Λ (9–15 cm), and Q (0.75–1.50 mL/h), were evaluated for the sorption of dodecane hydrocarbon (S_H, g/g) and motor oil (S_{MO}, g/g). Consequently, the chemometric models developed by means of the multiple regression method were used as two objective functions for process optimization, as given by:

$$\widehat{S}_H = 4.549 - 0.079U - 1.045\Lambda + 7.009Q + 0.11U\Lambda - 0.458UQ + 0.192\Lambda Q \quad (1)$$

$$\widehat{S}_{MO} = -81.617 + 4.415U + 4.538\Lambda + 44.631Q - 0.131U\Lambda - 2.235UQ \quad (2)$$

subjected to: $20 \leq U \leq 25$ (kV); $9 \leq \Lambda \leq 15$ (cm); $0.75 \leq Q \leq 1.50$ (mL/h)

where $\widehat{S}_H$ and $\widehat{S}_{MO}$ is the estimated sorption capacity (by models) for dodecane and motor oil, respectively. Note that the chemometric models developed in equations (1) and (2) correspond to first-order models including an offset, linear main effects, and a two-factor interaction term. For process optimization, the *desirability function approach* [10] was adopted. According to this method, a global desirability value (D) is defined as the geometric mean of two individual desirability functions, d_1 and d_2, obtained by mapping the objective functions $\widehat{S}_H$ and $\widehat{S}_{MO}$ into the normalized interval (0–1). Since the objective for this application is to maximize the sorption capacities, the optimization problem was formulated as a maximization task. Hence, the global desirability for optimization was expressed as:

$$D = \sqrt{d_1(\widehat{S}_H) \times d_2(\widehat{S}_{MO})} \rightarrow \max! \quad (3)$$

The numerical optimization based on the desirability function approach indicated the following optimal conditions for electrospinning parameters: $U^* = 25$ kV, $\Lambda^* = 15$ cm, and $Q^* = 0.75$ mL/h, for which the desirability value of $D = 0.966$ was obtained. These optimal conditions corresponded to experimentally determined sorption values of $S_H = 26.6$ g/g and $S_{MO} = 39.06$ g/g for the polysulfone fibrous material produced by electrospinning [2]. Therefore, the same optimal conditions were used for producing PSF-based composites [2, 3], PVDF-based materials [3, 7], and, more recently, for electrospinning of the copolymer poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) reported in this work.

The results for the system (PVDF-HFP) investigated herein are highlighted in Figure 2, showing images of (a) the experimental setup, (b) general aspects of the produced PVDF-HFP electrospun membrane, (c) water droplets onto the membrane surface, (d) SEM image, (e) EDAX spectrum, (f) FT-IR spectrum, and (g) mechanical behaviour. Hence, by the electrospinning of PVDF-HFP fibers (Figure 2a), a porous-fibrous material with the membrane aspect (Figure 2b) was obtained, exhibiting a porosity of 86.02 ± 0.36% determined by the gravimetric method using IPA as the liquid to fill the pores.

The shapes of water droplets onto the membrane surface (Figure 2c) highlighted the hydrophobic properties with a water contact angle of 130.8 ± 0.6°. The recorded SEM image (Figure 2d) of the electrospun membrane revealed a fibrous-porous scaffold with average fiber diameters of 0.24 ± 0.07 μm and inter-fiber voids ranging from 0.1 to 1.17 μm. It is worth noting that electrospinning the PVDF-HFP copolymer from a dope solution, with DMF: acetone (1:1) mix solvent, yielded ultrathin fibers that tended to align and aggregate parallel to the fiber axis. Within such fiber clusters, capillary forces might be enhanced, leading to improved liquid retention. The EDAX spectrum (Figure 2e) confirmed the presence of the expected elements (C and F), along with oxygen attributed to water molecules from the ambient humidity adsorbed on the fiber surface. The FT-IR spectrum (Figure 2f) recorded for the electrospun fibers is typical of PVDF-HFP copolymer. According to Figure 2f, the FT-IR spectrum of the electrospun PVDF-HFP fibers shows the β crystalline phase of PVDF (peaks: 841, 1277 cm^{-1}) together with the α crystalline phase (762 cm^{-1}), thus the electrospun membrane contains a mixture of PVDF crystalline phases. The band at 1180 cm^{-1} is a good indicator of the presence of the HFP group (CF_3). The valence and deformation bands for the $>CH_2$ group (2852, 2924, 1402 cm^{-1}) are also present, as expected for the PVDF chain.

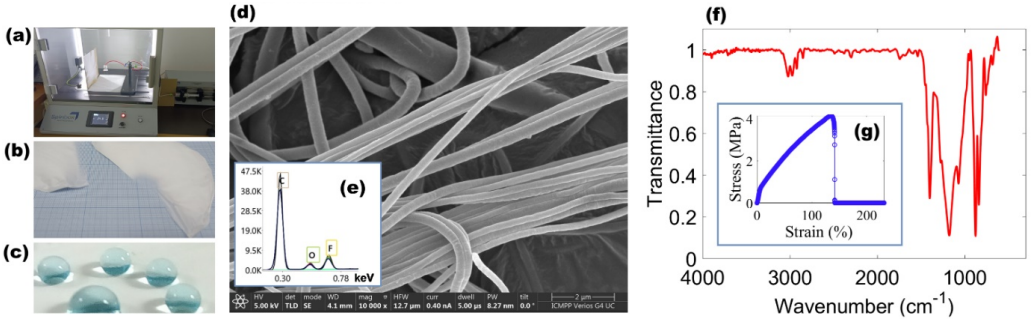

Figure 2. Preparation and characterization of the copolymeric electrospun membrane: (a) electrospinning setup used to prepare the fibrous material; (b) macroscopic appearance of the electrospun membrane; (c) water droplets on the membrane surface; (d) SEM image; (e) EDAX spectrum; (f) FT-IR spectrum; and (g) mechanical behavior

From the curve describing the mechanical properties (Figure 2g), plotted in the co-ordinates of the nominal stress (MPa), or the tensile strength at break, as a function of elongation/stretching (%), the Young's modulus for 5% elongation was determined to be 6.16 MPa, suggesting a moderate stiffness. Also, an elongation of 135% at break

was observed (Figure 2g), indicating a flexible membrane. These results suggested good mechanical properties for the electrospun material.

The produced electrospun fibrous-porous material (Figure 2b) was further tested for oil sorption, disclosing relevant sorption capacities of 9.3 g/g and 16.2 g/g for the uptake of motor oil (0.843 g/mL, 67 cP) and cooked palm oil (0.921 g/mL, 138 cP), respectively. The recovery of oils (70–84%) from the spent sorbent was possible by means of the centrifugation technique. Table 1 summarizes the performance of fibrous polymeric materials in oil sorption applications, providing a comparison between textile-derived PESt microfibers coated with polysiloxane and electrospun submicronic (or nanometric) fibers based on PSF, PVDF, and PVDF-HFP (co)polymers.

Table 1. Performance of various polymer-based fibrous materials for oil sorption applications

Fibrous sorbent (textile *vs.* electrospun fibers), [Ref.]	**Morphology of adsorbent (∅ - fiber diameter; V_{IF} – interfiber voids)**	**Oil type**	S **(g/g)**	**Recovery of oil, %**
Textile fibers PESt [4]	∅=10–50 μm, V_{IF}=2–230 μm	Motor oil	10.0	95–97
		Dodecane	5.5	95–97
Polysulfone (PSF) [2]	∅=0.05–1.70 μm, V_{IF}=0.5–15.0 μm	Motor oil	39.06	66–71
PSF/Nanoclay (NC) [3]	∅=0.40–2.10 μm, V_{IF}=1.80–2.60 μm	Motor oil	15.1	65–75
PSF/NC/$CoFe_2O_4$ [3]	∅=0.40–2.10 μm, V_{IF}=1.80–2.60 μm	Motor oil	27.0	65–75
PVDF/Nanoclay (NC) [3]	∅=0.20–1.20 μm, V_{IF}=2.80–3.40 μm	Motor oil	7.5	65–75
PVDF/NC/$CoFe_2O_4$ [3]	∅=0.20–1.20 μm, V_{IF}=2.80–3.40 μm	Motor oil	7.6	65–75
PVDF [7]	∅=0.11–1.18 μm, V_{IF}=0.29–21.80 μm	Motor oil	20.0	-
PVDF/$CoFe_2O_4$ [7]	∅=0.11–1.18 μm, V_{IF}=0.29–21.80 μm	Motor oil	16.09	-
PVDF-HFP [*this study*]	∅= 0.24±0.07 μm, V_{IF}=0.1..1.17 μm	Motor oil	9.3	70
		Cooked oil	16.2	84

According to Table 1, electrospinning can produce nonwovens with higher oil sorption capacities than conventional hydrophobic textile nonwoven sorbents. This enhanced performance can be correlated to the smaller fiber diameters of electrospun fibers (0.05–2.10 μm) compared to textile fibers (10–50 μm), which provide a greater surface contact area. On the other hand, oil recovery from spent sorbents by centrifugation is higher for textile nonwovens (95–97%) than for electrospun nonwovens (65–84%). This might be attributed to the larger inter-fiber voids or capillary pores in textile materials (2–230 μm) compared to those in electrospun nonwovens (0.1–21.8 μm). Hence, the production of advanced fibrous materials by electrospinning remains of significant interest, because their fibrous–porous morphology can be controlled by adjusting the operating parameters or the composition of the dope solution. Moreover, the incorporation of various fillers can impart additional functionalities, such as magnetic or oleophilic properties, or enhance the mechanical behavior of the materials.

3. Conclusions

In summary, this paper reports the fabrication of a fibrous-porous scaffold obtained through the electrospinning of the copolymer poly(vinylidene fluoride-co-hexafluoropropylene) under the following operating parameters: 25 kV applied voltage, 15 cm needle-to-collector distance, and 0.75 mL/h feed flow rate. The resulting hydrophobic material exhibited a porosity of 86.02±0.36%, and it was characterized by FT-IR, EDAX, SEM, as well as mechanical behaviour. Moreover, its application for oil sorption unveiled a relevant sorption capacity of 9.3 g/g for the uptake of motor oil. The feasibility of oil recovery from the spent sorbent was also demonstrated through centrifugation. The sorption performance of the produced electrospun nonwoven was compared with the capacities of other fibrous-porous materials reported in the literature.

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References

[1] KANDIYIL, J.; PAROLY, S.,;ATHIYANATHIL, S.; CHINGAKHAM, C. Electrospun nanofibrous membranes based sorbents for oil/water separation: Blends, composites, functionalized, and smart membranes – A mini review. *Journal of Environmental Chemical Engineering*. 2025, vol. 13, no. 3, pp. 1–28. ISSN 2213-3437. DOI: 10.1016/j.jece.2025.116262.

[2] COJOCARU, C.; PASCARIU DORNEANU, P.; AIRINEI, A.; OLARU, N.; SAMOILA, P.; ROTARU, A. Design and evaluation of electrospun polysulfone fibers and polysulfone/$NiFe_2O_4$ nanostructured composite as sorbents for oil spill cleanup. *Journal of the Taiwan Institute of Chemical Engineers*. 2017, vol. 70, pp. 267–281. ISSN 1876-1070. DOI: 10.1016/j.jtice.2016.11.005.

[3] PASCARIU DORNEANU, P.; COJOCARU, C.; SAMOILA, P.; OLARU, N.; AIRINEI, A.; ROTARU, A. Novel fibrous composites based on electrospun PSF and PVDF ultrathin fibers reinforced with inorganic nanoparticles: Evaluation as oil spill sorbents. *Polymers for Advanced Technologies*. 2018, vol. 29, pp. 1435–1446. ISSN 1042-7147; 1099-1581. DOI: 10.1002/pat.4255.

[4] COJOCARU, C.; PRICOP, L.; SAMOILA, P.; ROTARU, R.; HARABAGIU, V. Surface hydrophobization of polyester fibers with poly(methylhydro-dimethyl)siloxane copolymers: Experimental design for testing of modified nonwoven materials as oil spill sorbents. *Polymer Testing*. 2017, vol. 59, pp. 377–389. ISSN 0142-9418. DOI: 10.1016/j.polymertesting.2017.02.024.

[5] ZĂVOIANU, I. *Hidrologie*. Bucureşti: Editura Fundaţiei "România de Mâine", 1999. 319 p. ISBN 973-582-242-3.

[6] ASHRAF, R.; SOFI, H.S.; MALIK, A.; BEIGH, M.A.; HAMID, R.; SHEIKH, F.A. Recent trends in the fabrication of starch nanofibers: Electrospinning and non-electrospinning routes and their applications in biotechnology. *Applied Biochemistry and Biotechnology*. 2019, vol. 187, no. 1, pp. 47–74. ISSN 0273-2289, 1559-0291. DOI: 10.1007/s12010-018-2797-0.

[7] PASCARIU DORNEANU, P.; COJOCARU, C.; OLARU, N.; SAMOILA, P.; AIRINEI, A.; SACARESCU, L. Electrospun PVDF fibers and a novel PVDF/$CoFe_2O_4$ fibrous composite as nanostructured sorbent materials for oil spill cleanup. *Applied Surface Science*. 2017, vol. 424, pp. 389–396. ISSN 0169-4332. DOI: 10.1016/j.apsusc.2017.01.177.

[8] HAIDER, A.; HAIDER, S.; KANG, I.-K. A comprehensive review summarizing the effect of electrospinning parameters and potential applications of nanofibers in biomedical and biotechnology. *Arabian Journal of Chemistry*. 2015, vol. 11, no. 8, pp. 1165–1188. ISSN 1878-5352. DOI: 10.1016/j.arabjc.2015.11.015.

[9] OKUTAN, N.; TERZI, P.; ALTAY, F. Affecting parameters on electrospinning process and characterization of electrospun gelatin nanofibers. *Food Hydrocolloids*. 2014, vol. 39, pp. 19–26. ISSN 0268-005X. DOI: 10.1016/j.foodhyd.2013.12.022.

[10] BEZERRA, M.A.; SANTELLI, R.E.; OLIVEIRA, E.P.; VILLAR, L.S.; ESCALEIRA, L.A. Response surface methodology (RSM) as a tool for optimization in analytical chemistry. *Talanta*. 2008, vol. 76, no. 5, pp. 965–977. ISSN 0039-9140; 1873-3573. DOI: 10.1016/j.talanta.2008.05.019.

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