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nafion-few-layer graphene

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The proton-exchange polymer Nafion has practical applications in various fields of science and technology, such as electrochemical systems, ion-exchange water purification systems, production of ultrapure materials, and more. The durability and stability of this polymer are important for expanding its application range. Nafion/few-layer graphene composites demonstrated enhanced thermal stability of the proton-exchange polymer Nafion compared to the pure material. The gravimetric method was used to study the free-radical attack of $-$OH radicals formed in the Fenton reaction on substrates made of different materials (silicone, polyethylene, glass, silicon) and composite films of Nafion/few-layer graphene on them. Based on experiments and statistical analysis of the experimental data array, the conditions (substrate material, error margin, sensitivity) for the applicability of the stability measurement method for the composite via mass loss were determined. The data of the study of the stability of Nafion/few-layer graphene composites of different component compositions are presented.

Keywords: Sulfopolymer, gravimetry, Fenton reaction, stableness.

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Introduction

Ion-exchange polymer materials of the Nafion type, including its modifications, have been widely and long used for various technical purposes [1]. The most well-known areas of use include:

1) Membranes in energy storage devices, such as in flow batteries [2];

2) Membranes in the chemical industry for the separation of impurities in the production of semiconductors and high purity materials [3];

3) Acid catalysts [4];

4) Waste management: Nafion membranes reduce waste during maintenance and cleaning of electroplating baths, as well as during recovery of waste solutions and washing waters [5];

5) Use in harsh conditions of chlorine and alkali production: Nafion membranes provide high efficiency of the electrochemical process, high reliability of production, high product quality [6];

6) Ion exchange: Nafion granules serve as long-lasting perfluorinated cation exchange resins that, unlike other non-fluorinated resins, can operate under extreme temperatures and chemical environments [7];

7) Membranes and proton-conducting medium in hydrogen energy [8], in water electrolyzers for the production of hydrogen and oxygen [9];

8) Application in biosensors [10];

9) The use of Nafion as an antimicrobial supplement [11].

It is obvious that the resistance of Nafion to various influences plays an important role in the practical application of the polymer and the materials containing it [12–14]. One of the types of such effects is the free radical attack of a polymer in electrochemical devices due to the formation and decomposition of hydrogen peroxide or for other reasons [15,16]. To assess the resistance of polymer membranes to this effect (free radical attack), it is customary to use the Fenton reaction [17], which results in the formation of free radicals $-$OH, which act on the tested material. In this case, the degree of degradation of Nafion is usually determined by the concentration of the fluoride ion converted into a solution as a result of decomposition. For this purpose, the potentiometric method with a fluorine-selective electrode or other physical and physico-chemical detection methods are often used [18–21]. Most of the works published in the scientific literature are devoted to the study of Nafion degradation in the form of membranes. Accelerated testing techniques are relatively well developed for such facilities. At the same time, for composite materials containing Nafion as one of the components, such a direction has been developed significantly less. The specific feature of such objects is the presence of components other than Nafion. These components can also be corroded or simply emit from the composite when the Nafion is destroyed. The assessment of the stability of such composites requires other approaches, since measuring the concentration of F^--ions gives an idea only of the fraction of decomposed polymer (Nafion), i.e., only one of the components of the composite, and does not reflect the overall picture. The method does not allow us to determine how much of the rest of its components is washed out of the composite, i.e. how the entire composite collapses.

One of the approaches to assessing the stability of a composite is to measure the loss of its mass during treatment with Fenton's reagent. Combining these two approaches (measuring fluorine emission and weight loss) it allows a comprehensive assessment of the ongoing degradation. In the scientific literature, studies of the stability of proton-exchange polymers using gravimetry are mainly focused on bulk membranes with sufficient mass [22–24]. The method involves weighing the sample before and after processing. Since in these cases the mass of the object of study can be quite large, there are no special difficulties in performing the method and in obtaining reliable values of mass loss (they are well measured due to their sufficient magnitude). When it comes to composites, multicomponent systems, two question arise that need to be resolved before using the gravimetry method. The first question is how to ensure the solidity of the studied composite sample during the experiment? The second question is how to ensure a sufficiently low error in the results when using small amounts of composite?

For studies of small quantities of composite materials, it is advisable to form monolithic films on inert substrates, which will ensure convenience in handling such structures. At the same time, in order to ensure good reproducibility of experimental data on the weight loss of the composite film, the effect of an aggressive medium on the substrate should be either insignificant in relation to the effect on the composite film, or precisely controlled. In addition, the substrate material must ensure the formation of a uniform composite film, the film must have high adhesion to the substrate and not peel off during the experiment. It is desirable that the substrate does not contain fluorine so that a combined method can be used, including measuring the mass loss and concentration of fluoride ions transferred to the solution.

The Nafion-graphene composite (G) is interesting for its thermal stability [12]. In this composite, the Nafion polymer begins to lose sulfogroups at significantly higher temperatures than in its pure form. However, there are practically no studies of composite's resistance to radical attacks in scientific publications.

The purpose of this study was to establish the conditions (substrate material, film weight, sensitivity, and error) of the applicability of the composite stability measurement method by weight loss and to study the stability of Nafion-G-Pt/C composites of various component compositions.

1. Materials and methods

The following reagents and materials were used as precursors for the preparation of the composite:

1) Nafion DE1021 aqueous solution (DuPont$^{\text{TM}}$, Wilmington, Delaware, USA);

2) isopropanol (99.80 %, JSC „EKOS-1", Russia);

3) Thermally expanded graphite (TEG) (NPO „Graphene Materials" LLC, Russia), obtained using the technology described in the patent [25];

4) Platinum carbon black E-TEK (40 % Pt) [26].

To test chemical stability, a 10 % solution of hydrogen peroxide was used, prepared from 30 % solution of hydrogen peroxide grade 8–4 („Lega", Russia) by dilution with water. Deionized water with a resistivity at room temperature $\rho \geq 18\,\text{M}\Omega \cdot \text{cm}$ was used.

Table 1. Some characteristics of the substrates used to form films of Nafion-G composite material

Material	Size, cm^2	Thickness, μm	Weight, g
Polyethylene	3.08 (14×22(±0.5) mm)	35	0.0076–0.012
Glass	17.5 (26×76(±0.5) mm)	1000	4.80–4.85
Silicon	3.08 (14×22(±0.5) mm)	350	0.210–0.230
Silicone	3.08 (14×22(±0.5) mm)	480	0.177–0.180

2. Characteristics of media for film formation of the studied materials

The following materials have been tested as substrates for film formation: silicon wafers (Si KDB 7.5(100)), 76 × 26 mm slides of the TEGET type, polyethylene film (high-pressure polyethylene), silicone film.

Table 1 shows the characteristics of the media used to form films of the studied materials.

3. Preparation of the dispersion

Dispersions of the Nafion-G and Nafion-G-Pt/C systems with different component ratios (Table 2) defined set gravimetrically were prepared for the study.

2–8 mg of TEG, estimated amount mg 2 % of the working solution of Nafion (the working solution was obtained by dilution of 20 % commercial solution in deionized water), calculated amount of Pt/C (E-TEK 40 % Pt) (if required), 10 cm^3 of isopropanol–water mixture, taken in a ratio of 1 : 1, were taken in a 10 cm^3 polystyrene tube with cap. The tube was closed with a cap, placed in a Branson 3510 ultrasonic bath, and was subjected to ultrasonic dispersion until a homogeneous dispersion was obtained that did not delaminate for 3 min. A portion (1 drop) of the dispersion was periodically sampled for studies of the dispersion process by ultraviolet (UV) spectroscopy.

4. Preparation of samples for research

The substrates were pre-washed in isopropyl alcohol, and the mixtures were dispersed for 15 min before application.

Table 2. Compositions of the studied composites

Initial composition: Nafion:TEG:Pt/C, %	Resulting composite
100 : 0 : 0	Nafion
20 : 80 : 0	Nafion-G
50 : 50 : 0	
80 : 20 : 0	
10 : 45 : 45	Nafion-G-Pt/C
14 : 56 : 30	
35 : 35 : 30	
40 : 30 : 30	
46 : 24 : 30	
60 : 20 : 20	
80 : 10 : 10	

A dispersion of a given composition was pipetted drop by drop onto the surface of a substrate thermostatically controlled on a plate of the heating brand „C-MAG HP 7“ at 80 °C–85 °C. The next portion was applied after the previous one had dried. A layer with a weight of 2–3 mg was formed. The amount of applied material was controlled gravimetrically.

To apply the film, a portion of the dispersion was taken and diluted approximately 1:3 with an isopropanol–water mixture (1 : 1 by volume). During application, an uncoated edge was left on the substrate to handle it with tweezers.

5. Measurement procedure

5.1. Microscopic studies

Optimization of analytical measurements was carried out within the framework of the previously described approach [27]. Before imaging, the dispersion of the samples was dried on a copper mesh with a diameter of 3 mm, which were fixed in a special holder. The microstructure of the samples was studied by scanning transmission electron microscopy with field emission (FE-STEM) on a Hitachi SU8000 electron microscope. The images were recorded in bright-field scanning transmission electron microscopy (bright field mode) mode at an accelerating voltage of approximately 30 kV.

5.2. Recording of UV-visible absorption spectra of dispersions

The absorption spectra were recorded in a quartz cuvette with an absorption layer length of 1 cm using a two-beam Specord 210 type spectrophotometer (Analytik Jena) with WinAspect software. A mixture of isopropanol–water taken in a volume ratio of 1 : 1 was used as the reference solution. The following conditions for recording the spectra were used: step 0.1 nm; scanning speed 5 nm/s. The spectral width of the slit was 1 nm. The light source was switched from a deuterium lamp to an incandescent lamp at 370 nm. The measurements were carried out at room temperature $\sim 25\,^{\circ}$C and atmospheric pressure.

5.3. Study of resistance to radical attacks

Repeated treatment with Fenton's reagent of pure substrates was used to study the possibilities of the method and the choice of a substrate for the formation of films of composite material. Composite films were formed on a selected substrate at the second stage of the work.

The samples were treated in Fenton's reagent as follows. A Fenton solution was prepared, as described in Ref. [28] immediately before the experiment. To do this, a 0.2 cm^3 solution of Mohr salt with a concentration of 7 g/l was taken into a measuring cylinder with a capacity of 50 cm^3. 40 cm^3 of distilled water were added, followed by 5 cm^3 of 30 % solution of H_2O_2, and the solution was made up to a final volume of 50 cm^3. Thus, 50 cm^3 3 % solution of H_2O_2 containing 4 ppm Fe was obtained.

To study the stability to radical attacks, a freshly prepared Fenton solution was poured into a glass-carbon beaker, it was thermostated at 80 °C and samples were placed in it for 2 h. After that, the samples were washed with water and dried in air. The samples were weighed on analytical scales Mettler Toledo XP 205 („Mettler-Toledo International Inc“, Greifensee, Switzerland).

6. Calculations

6.1. Calculation of porosity

The thickness of the films was measured with a thickness gauge with a resolution of 0.5 μm at 10 layer points. The resulting thickness value was taken as the arithmetic mean. The area of the electrodes was 1 cm^2. The porosity of the films was calculated based on the known component composition, thickness and area by the ratio

$$P = 1 - \frac{\Sigma_{i=0}^{n} \frac{\omega_i G}{d_i}}{V}, \qquad (1)$$

where P is the porosity (volume fraction of pores) of the film; V is the volume of the film (in this case, with a length and width of the film of 1 cm, the volume is numerically equal to the thickness); ω_i is the mass fraction of ith component; G is the mass of electrode; d_i is the density of ith component.

6.2. Statistical processing of results

The standard deviation (SD) and the standard error (SE) were calculated using known ratios

$$SD = \sqrt{\frac{\Sigma(x_i - \mu)^2}{N}}, \tag{2}$$

$$SE = SD/\sqrt{N}, \tag{3}$$

where x_i is the current weight loss value; μ is the average weight loss; N is the sample size.

The film sample weight loss Δm on the substrate was calculated based on the total weight loss Δm_{sum} of the substrate with the film and the preset weight loss of the pure (without film) substrate at a similar processing time in Fenton's reagent Δm_{sub}:

$$\Delta m = \Delta m_{sum} - \Delta m_{sub}/2. \tag{4}$$

7. Discussion of the results

7.1. UV spectroscopy

UV spectroscopy of was used to control the process of TEG peeling in the prepared dispersions [29–32].

Fig. 1 shows the characteristic absorption spectrum of a sample of Nafion-G dispersion after ultrasonic treatment.

It can be seen from the figure that the spectrum contains absorption peaks at ~ 230, ~ 265 and ~ 310 nm, characteristic of low-layer G and its oxide [32]. The shorter-wavelength peak is responsible for the $\pi \to \pi^*$ transition, while the longer-wavelength (310 nm) is responsible for $n \to \pi^*$. The peak at ~ 265 nm is characteristic of the reduced oxide of G GO); it is noted in Ref. [29] that there is a peak at 235 nm on the absorption spectra of the aqueous dispersion of GO in the UV-visible region, which corresponds to $\pi \to \pi^*$ electronic junction of sp^2-bonds C=C. This peak shifts towards a longer wavelength (265 nm) after the restoration of GO to G. This effect is explained by an increase in the π-conjugation [30]. Less energy is required for the transition as the π-conjugation increases, which corresponds to the observed shift of absorption to a longer wavelength region.

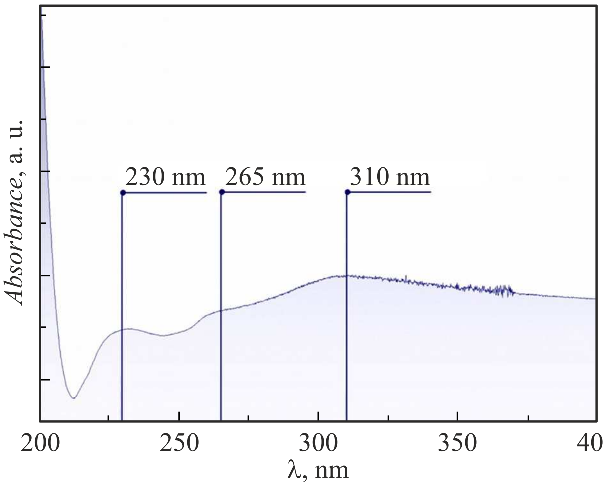

Figure 1. Absorption spectrum of Nafion-G dispersion in isopropanol-water medium in 1 : 1 volume ratio; concentration of Nafion 0.33 g/l.

The article [31] shows that variances containing a low-layer (1–3 layers) GO can be distinguished from variances containing a multi-layer (4–10 layers) and GO with a large number of layers (> 10) by the intense peak in their UV-visible spectra at 230 nm. GO with 1–3 layers has a single peak, and GO with 4–10 layers has a weak peak in the form of a shoulder. As the number of layers increases, the shoulder intensity of the multilayer GO tends to decrease. There is no peak or shoulder for GO with a large (> 10) number of layers. In our case, the peak at 230 nm is well expressed. Thus, our system contains a mixture of G and partially oxidized G (i.e., GO) with a small number of layers of (1–3).

7.2. Formation of films of composite material of the Nafion-G-Pt/C system on various substrates

Fig. 2 shows microimages of the composite of the Nafion-G-Pt/C system with varying degrees of magnification. The dried sample (Fig. 2, *a*) is a porous material consisting of separate agglomerates. Particles of graphene material with a lateral size of the order of 50 μm are visible with a higher magnification (Fig. 2, *b*). Fig. 2, *c* shows spherical particles of platinum carbon black located on a graphene sheet.

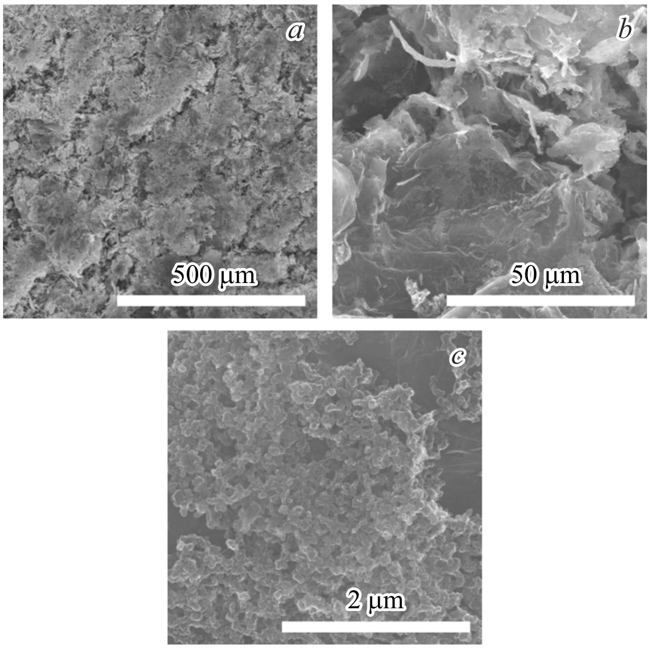

Figure 2. Microimages of the Nafion-G-Pt/C composite of the initial composition 40 : 30 : 30 (it a–c).

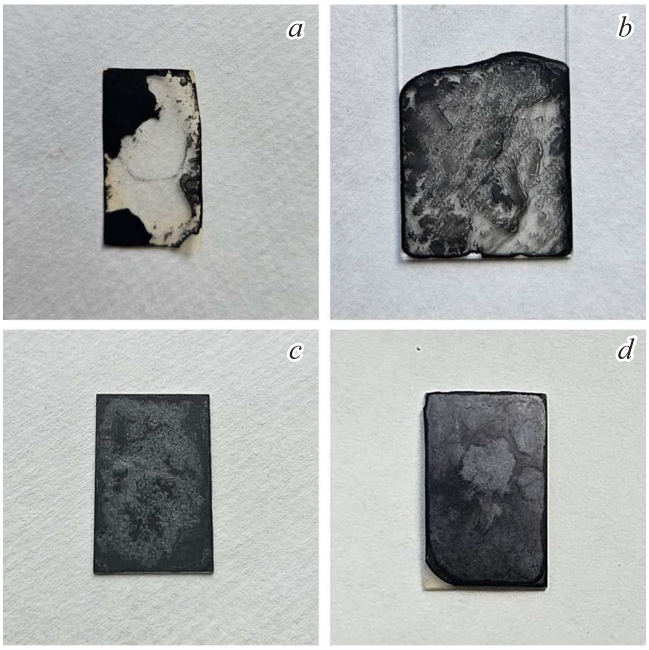

Figure 3. Appearance of Nafion-G composite samples on various types of substrates: *a* — polyethylene; *b* — glass; *c* — silicon; *d* — silicone.

Composite films on various substrates are shown in Fig. 3. The samples on a polyethylene substrate (Fig. 3, *a*) were structures with alternating areas covered and uncovered with the material. The samples on the glass demonstrated delamination of the dispersion with segregation of G particles in the form of separate clusters along the edges of the coating area, the films turned out to be visually heterogeneous (Fig. 3, *b*). The films on the surface of the silicone substrate (Fig. 3, *d*) formed visually uniformly and had good adhesion (they did not peel off during subsequent processing in Fenton's reagent). The formation of composite films on the silicon surface initially led to the formation of an island structure, which subsequently merged into a single film with an increase in the amount of applied material (Fig. 3, *c*).

During the subsequent processing of silicon samples in Fenton's solution, the composite film peeled off from the substrate, which made it impossible to measure the loss of sample weight after processing. In this regard, silicon and glass substrates were rejected as not providing uniformity of the studied samples and the possibility of measuring mass loss due to radical attack of the material.

7.3. Dependence of porosity of Nafion-G-Pt/C composite samples on Nafion content

Individual samples of composites of different compositions were made in order to study the dependence of the porosity of the samples on the percentage of Nafion in them. Table 3 summarizes the data on the porosity of samples of the Nafion-G-Pt/C system. The values given in Table 3 indicate that the Nafion polymer strongly affects porosity. As the polymer content increases, monolithic structures containing a very small number of pores are formed. Such structures are obviously difficult to access for radical attacks in the thickness of the film and are attacked only from the surface.

Table 3. Data on the porosity of composite materials

Initial composition of Nafion : TEG : Pt/C, %	Porosity, %
10 : 45 : 45	62
40 : 30 : 30	59
60 : 20 : 20	16
80 : 10 : 10	< 5

In the case of a relatively small proportion of Nafion in the composite (the first and second samples with a polymer fraction of 10 and 40 %), the porosity is quite large and amounts to 62 and 59 %, respectively. This makes the highly developed inner surface of the film accessible to the Fenton reagent.

7.4. Stability study of polyethylene and silicone substrates

Fig. 4 shows photographs of the substrates before and after treatment in Fenton's reagent.

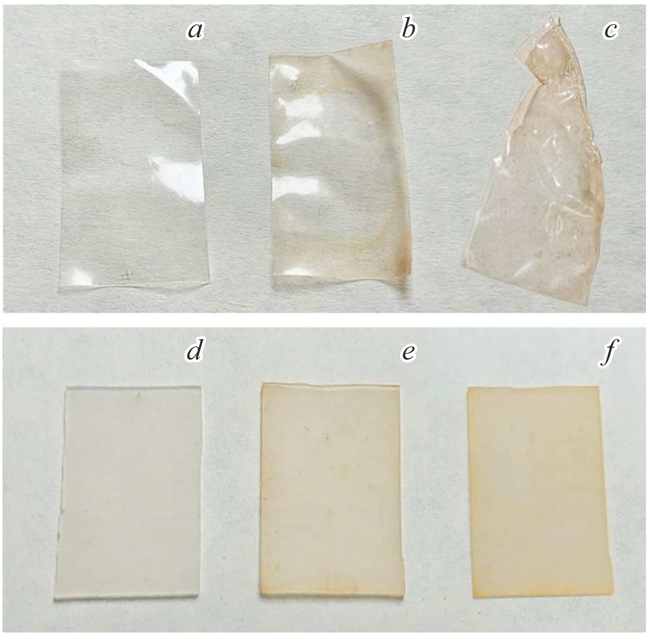

Figure 4. Appearance of the substrates after treatment in Fenton's reagent. Polyethylene: *a* — before treatment; *b* — after one treatment; *c* — after five treatments. Silicone: *d* — before treatment; *e* — after one treatment; *f* — after five treatments.

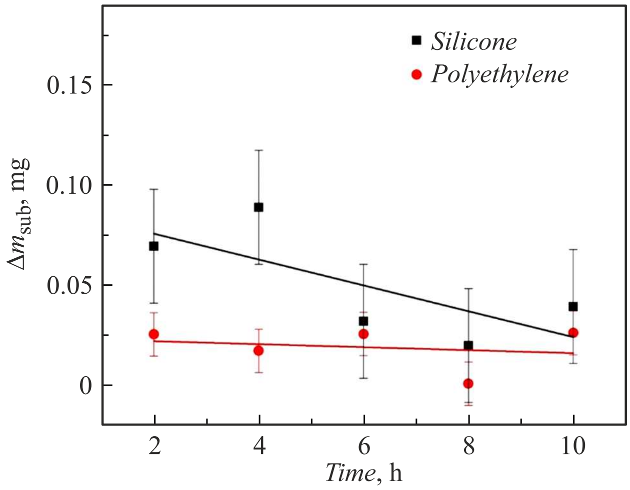

Figure 5. Dependences of the decrease in the mass of polyethylene and silicone substrates and on the number of treatment steps with Fenton's reagent. The duration of one processing step is 2 h.

The images show that the polyethylene substrate has deteriorated significantly, while the original shape of the silicone substrate has remained virtually unchanged after processing. At the same time, the color of both substrates changed, and a yellowish tint appeared, which indicates a noticeable effect of Fenton's reagent on both substrates.

To assess the sensitivity of the method used and the reproducibility of the results obtained, a series of parallel experiments were conducted on the processing of substrates in Fenton's reagent.

Data on the weight loss of the substrates as a result of treatment in Fenton's reagent are shown in Fig. 5.

The dependences of the decrease in the mass of the substrates (Fig. 5) are approximated by straight lines with a slight negative slope. At the same time, the initial value and slope are higher for silicone substrates. This can be explained by their greater thickness and mass. With a ten-hour total treatment, the weight loss of these two materials is practically comparable.

The distribution curves of the mass loss values (Δm_{sub}) in the data arrays of multiple parallel experiments are shown in Fig. 6. It can be seen from the figure that the random variable Δm has a distribution close to normal. This makes it possible to use variance statistics in relation to it.

Table 4 shows the results of statistical processing of mass loss data after repeated exposure to Fenton's reagent for two types of substrates.

The „three sigma“ criterion was used to evaluate the sensitivity of the method. Fig. 7 shows the dependences of three standard deviations on the number of substrate treatments with Fenton's reagent. The points are well approximated by straight lines.

Dependencies 3SD(N) have a slight negative slope as follows from Table 4 and Fig. 7. In the case of silicone substrates, the slope is greater, i.e., the sensitivity of the method when using both polyethylene and silicone substrates increases slightly with an increase in the number of pretreatments. The sensitivity was about $8 \cdot 10^{-5}$ g during five preliminary treatments of the substrates. This is probably due to the leaching of the most active component of the substrate. It follows from this that pretreatment of empty substrates in Fenton's reagent is necessary to stabilize the results of the study and to obtain more reproducible data.

7.5. Study of stability of Nafion-G and Nafion-G-Pt/C composites

The appearance of the Nafion-G-Pt/C composite film before and after treatment with Fenton's reagent is shown in Fig 8. It can be seen that the film retains adhesion to the substrate after processing, but local deformations are present.

Table 5 and Fig. 9 show data on the destruction of films of various compositions as a result of exposure to Fenton's reagent.

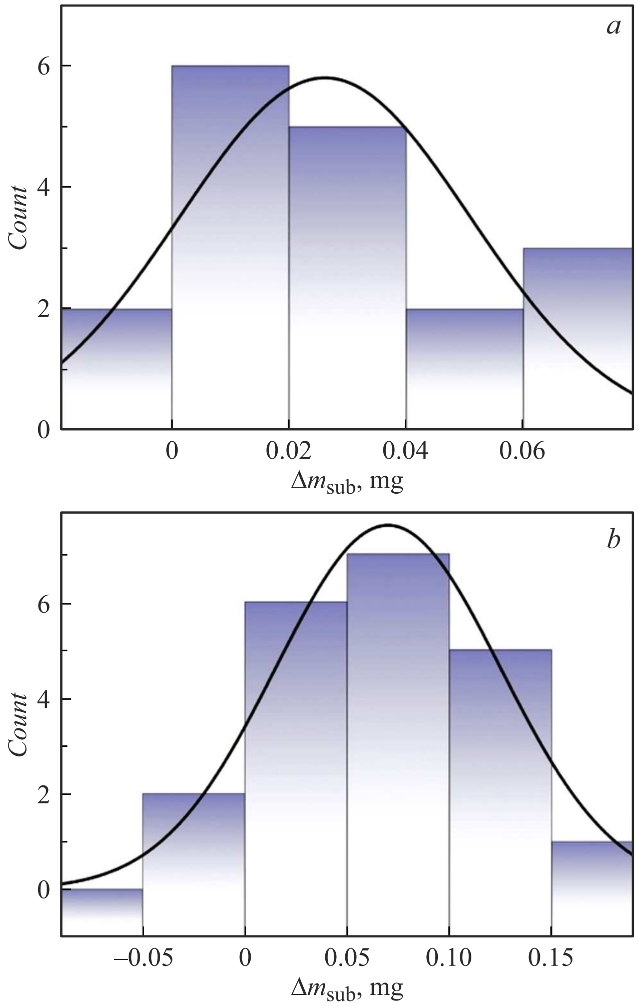

Figure 6. Distribution curves of the data sample (N ~ 20) by loss of substrate mass after the first treatment for 2 h in Fenton's reagent: *a* — polyethylene; *b* — silicone.

Table 4. Statistical characteristics of the data sample (N 20) decrease in the mass of the substrates during sequential treatment with Fenton's reagent for 2 h each

Sample	Treatment No.	N	Weight Loss, g	Standard Deviation (SD)	Standard Error (SE)	3SD, g
Polyethylene	1	18	$3 \cdot 10^{-5}$	$2 \cdot 10^{-5}$	$6 \cdot 10^{-6}$	$7 \cdot 10^{-5}$
	2	19	$2 \cdot 10^{-5}$	$2 \cdot 10^{-5}$	$5 \cdot 10^{-6}$	$6 \cdot 10^{-5}$
	3	19	$3 \cdot 10^{-5}$	$2 \cdot 10^{-5}$	$4 \cdot 10^{-6}$	$5 \cdot 10^{-5}$
	4	20	$2 \cdot 10^{-6}$	$9 \cdot 10^{-6}$	$2 \cdot 10^{-6}$	$3 \cdot 10^{-5}$
	5	19	$3 \cdot 10^{-5}$	$3 \cdot 10^{-5}$	$6 \cdot 10^{-6}$	$8 \cdot 10^{-5}$
Silicone	1	21	$7 \cdot 10^{-5}$	$6 \cdot 10^{-5}$	10^{-5}	$2 \cdot 10^{-4}$
	2	19	$9 \cdot 10^{-5}$	10^{-6}	$2 \cdot 10^{-5}$	$3 \cdot 10^{-4}$
	3	19	$3 \cdot 10^{-5}$	$4 \cdot 10^{-5}$	$8 \cdot 10^{-6}$	10^{-4}
	4	19	$2 \cdot 10^{-5}$	$2 \cdot 10^{-5}$	$6 \cdot 10^{-6}$	$7 \cdot 10^{-5}$
	5	19	$4 \cdot 10^{-5}$	$2 \cdot 10^{-5}$	$5 \cdot 10^{-6}$	$6 \cdot 10^{-5}$

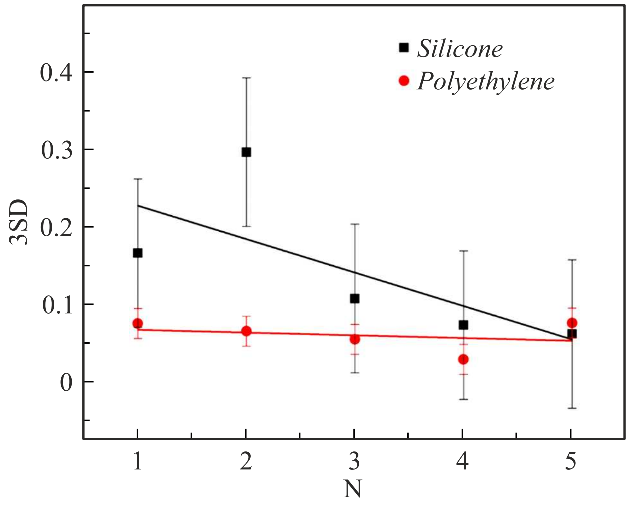

Figure 7. Dependences of the three standard deviations (3SD) of the mass loss during one treatment for 2 h on the number of treatments with Fenton's reagent for various substrates (polyethylene, silicone).

Figure 8. Photos of the Nafion-G-Pt/C composite film of the initial composition 14 : 56 : 30 before (*a*) and after (*b*) treatment with Fenton's reagent.

Materials with low and high Nafion polymer content have the greatest stability as follows from Table 5 and Fig. 9. The decrease of weight (the proportion of the destroyed film) is the greatest with a polymer content of about 50 %. This can be explained by the influence of the composite structure on vulnerability to Fenton's reagent. In the case of commensurate amounts of components, a porous structure is formed with good accessibility of the polymer surface to attacking radicals. With a low polymer content, even when completely destroyed, it makes a small contribution to the weight loss of the film.

It also follows from Fig. 9 that the content of platinum carbon black does not significantly affect the film's resistance to radical attacks. The rather strong point spread can be explained by small values of the analytical signal, the measured values of the mass loss are not far from the sensitivity of the method (~ 0.1 mg).

Conclusion

A gravimetric method for studying the resistance of small samples of composites based on a proton-exchange polymer of the Nafion type to free radical attacks is proposed.

Of the tested substrates, a silicone substrate is the most suitable for forming films of the Nafion-G-Pt/C composite system. It has the most stable form during exposure to Fenton's reagent and exhibits good adhesion to the composite film.

Gravimetric measurement of mass loss has a sensitivity limit of about 0.1 mg, and pretreatment of the substrate with

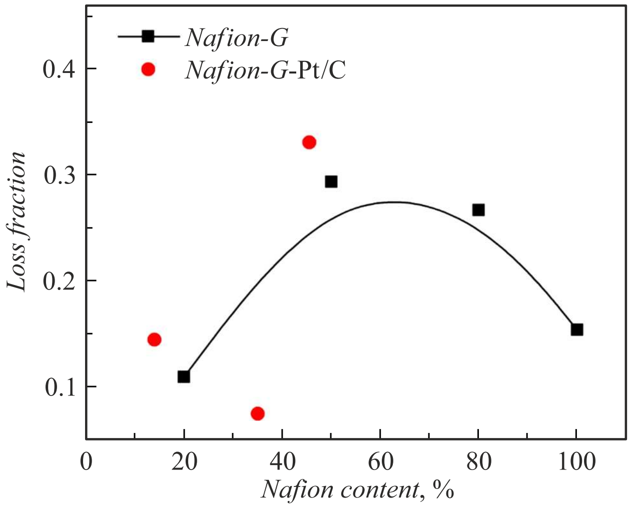

Figure 9. Dependence of the degree of destruction of composite films by Fenton's reagent on the content of Nafion polymer.

Table 5. Decrease in the mass of composite films of various compositions during treatment with Fenton's reagent

Initial composition Nafion : TEG : Pt/C, %	Weight of film, mg	Loss of weight films after treatment, mg	Proportion of loss of weight of film
100 : 0 : 0	2.28	0.35	0.15
20 : 80 : 0	2.2	0.24	0.11
50 : 50 : 0	2.15	0.63	0.29
80 : 20 : 0	2.29	0.61	0.27
14 : 56 : 30	2.15	0.31	0.14
35 : 35 : 30	2.15	0.16	0.074
46 : 24 : 30	2.18	0.72	0.33

Fenton's reagent leads to a decrease in the rate of mass loss of the substrate during processing and a decrease in its dispersion.

The studied composites of the Nafion-G-Pt/C system showed the following patterns. Materials with low and high Nafion polymer content demonstrate the greatest stability. The highest degree of film destruction is observed with a polymer content of about 50 %. This is because the composite's structure in this case makes it particularly susceptible to the effects of Fenton's reagent. When the content of the components is commensurate, a porous structure is formed, characterized by good accessibility of the polymer surface to attacking radicals. With a low polymer content, even when completely destroyed, it makes a small contribution to the weight loss of the film. The introduction of platinized carbon black does not significantly affect the film's resistance to radical attacks.

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Conflict of interest

The authors declare that they have no conflict of interest.

References

[1] C. Heitner-Wirguin. J. Membr. Sci., **120** (1), 1 (1996). DOI: 10.1016/03767388(96)00155-X

[2] F. Lulay, C. Weidlich, M. Valtiner, C.M. Pichler. Green Chem. Lett. Rev., **16** (1), 2274529 (2023). DOI: 10.1080/17518253.2023.2274529

[3] A.J. Seen, K.J. Cavell, A.M. Hodges, A.W.-H. Mau. J. Chem. Soc., Dalton Trans., **8**, 1381 (1992). DOI: 10.1039/dt9920001381

[4] K.-L. Huang, T.M. Holsen, J.R. Selman. J. Membr. Sci., **221** (1-2), 135 (2003). DOI: 10.1016/S0376-7388(03)00254-0

[5] M.E.I. Ahmed, K.-L. Huang, T.M. Holsen. Ind. Eng. Chem. Res., **48** (14), 6805 (2009). DOI: 10.1021/ie900149a

[6] R. Kuwertz, I. Gonzalez Martinez, T. Vidaković-Koch, K. Sundmacher, T. Turek, U. Kunz. Electrochem. Commun., **34**, 320 (2013). DOI: 10.1016/j.elecom.2013.07.035

[7] Electronic resource. *Nafion Resins: Novel Device Applications* Available at: https://www.sigmaaldrich.com/content/dam/sigma-aldrich/docs/Aldrich/Instructions/nafion_resins.pdf?srsltid=AfmBOorqZHjMz6VHFJGW_k7-Wq6xdHA1km-2IZKdvrj3LshIYk-L_hie

[8] S. Ahmad, T. Nawaz, A. Ali, M.F. Orhan, A. Samreen, A.M. Kannan, Int. J. Hydrog. Energy, **47** (44), 19086 (2022). DOI: 10.1016/j.ijhydene.2022.04.099

[9] H. Ito, T. Maeda, A. Nakano, H. Takenaka. Int. J. Hydrog. Energy, **36** (17), 10527 (2011). DOI: 10.1016/j.ijhydene.2011.05.127

[10] R.M. Kakhki. Int. J. Polym. Mater. Polym. Biomater., **73** (17), 1470 (2024). DOI: 10.1080/00914037.2023.2297436

[11] Y. Cheng, C.I. Moraru. Colloids Surf. B Biointerfaces, **162**, 16 (2018). DOI: 10.1016/j.colsurfb.2017.11.016

[12] A.O. Krasnova, N.V. Glebova, A.G. Kastsova, M.K. Rabchinskii, A.A. Nechitailov. Polymers, **15** (9), 2070 (2023). DOI: 10.3390/polym15092070

[13] A.A. Nechitailov, P. Volovitch, N.V. Glebova, A. Krasnova. Membranes, **13** (3), 342 (2023). DOI: 10.3390/membranes13030342

[14] N.V. Glebova, A.S. Mazur, A.O. Krasnova, I.V. Pleshakov, A.A. Nechitailov. Nanosystems: Phys. Chem. Math., **14** (2), 202 (2023). DOI: 10.17586/2220-8054-2023-14-2-202-207

[15] Z.A.R. Gautama, Y.A. Hutapea, B. Hwang, J. Matsuda, A. Mufundirwa, T. Sugiyama, M. Ariyoshi, S. Fujikawa, S.M. Lyth, A. Hayashi, K. Sasaki, M. Nishihara. J. Membr. Sci., **658**, 120734 (2022). DOI: 10.1016/j.memsci.2022.120734a

[16] F.C. Teixeira, A.P.S. Teixeira, C.M. Rangel. Int. J. Hydrog. Energy, **48** (96), 37489 (2023). DOI: 10.1016/j.ijhydene.2023.04.063

[17] K. Hongsirikarn, X. Mo, J.G. Goodwin, S. Creager. J. Power Sources, **196** (6), 3060 (2011). DOI: doi.org/10.1016/j.jpowsour.2010.11.133

[18] M. Aoki, H. Uchida, M. Watanabe. Electrochem. Commun., **8** (9), 1509 (2006). DOI: 10.1016/j.elecom.2006.07.017

[19] C. Chen, T.F. Fuller. Polym. Degrad. Stab., **94** (9), 1436 (2009). DOI: 10.1016/j.polymdegradstab.2009.05.016

[20] M. Inaba, T. Kinumoto, M. Kiriake, R. Umebayashi, A. Tasaka, Z. Ogumi. Electrochim. Acta, **51** (26), 5746 (2006). DOI: 10.1016/j.electacta.2006.03.008

[21] V.O. Mittal, H.R. Kunz, J.M. Fenton. J. Electrochem. Soc., **154**, B652 (2007). DOI: 10.1149/1.2734869

[22] H. Jang, T. Ryu, S.C. Sutradhar, F. Ahmed, K. Choi, H. Yang, S. Yoon, W. Kim. Int. J. Hydrog. Energy, **42** (17), 12768 (2017). DOI: 10.1016/j.ijhydene.2017.01.112

[23] F. Ahmed, S.C. Sutradhar, T. Ryu, H. Jang, K. Choi, H. Yang, S. Yoon, Md.M. Rahman, W. Kim. Int. J. Hydrog. Energy, **43** (10), 5374 (2018). DOI: 10.1016/j.ijhydene.2017.08.175

[24] T.S. Mayadevi, B.-H. Goo, S.Y. Paek, O. Choi, Y. Kim, O.J. Kwon, S.Y. Lee, H.-J. Kim, T.-H. Kim. ACS Omega, **7** (15), 12956 (2022). DOI: 10.1021/acsomega.2c00263

[25] V.I. Mazin, E.V. Mazin. *Sposob polucheniya poristogo uglerodnogo materiala na osnove vysokorasshcheplennogo grafita* (Patent RF № 2581382, publ. 20.04.2016) (in Russian)

[26] Electronic resource: *Pt electrocatalysts. 40 % Pt on Vulcan XC-72* (2025). Available at: https://premetek.com/product/40-pt-on-vulcan-xc-72/

[27] V.V. Kachala, L.L. Khemchyan, A.S. Kashin, N.V. Orlov, A.A. Grachev, S.S. Zalesskiy, V.P. Ananikov. Russ. Chem. Rev., **82** (7), 648 (2013). DOI: 10.1070/RC2013v082n07ABEH004413

[28] Electronic resource. *EU harmonized protofcols for testing of low temperature water electrolysis* (2021). Available at: https://op.europa.eu/en/publication-detail/-/publication/bbbeba00-ee82-11eb-a71c-01aa75ed71a1

[29] F.T. Johra, J.-W. Lee, W.-G. Jung. J. Ind. Eng. Chem., **20** (5), 2883 (2014). DOI: 10.1016/j.jiec.2013.11.022

[30] Y. Zhou, Q. Bao, L.A.L. Tang, Y. Zhong, K.P. Loh. Chem. Mater. **21** (13), 2950 (2009). DOI: 10.1021/cm9006603

[31] Q. Lai, S. Zhu, X. Luo, M. Zou, S. Huang. AIP Advances, **2** (3), 032146 (2012). DOI: 10.1063/1.4747817

[32] A.G. Kastsova, N.V. Glebova, A.A. Nechitailov, A.O. Krasnova, A.O. Pelageikina, I.A. Eliseyev. Tech. Phys. Lett., **48** (12), 61 (2022). DOI: 10.21883/TPL.2022.12.54950.19268

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