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PET TRACK-ETCHED MEMBRANES WITH DEPOSITED SILVER NANOTUBES AS A CATALYST FOR HYDROGEN PEROXIDE DECOMPOSITION

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Promoter – A.Mashentseva and J.Dzhakupova.

Here we report our recent research activities on the application of the Ag/PET composites for decomposition of hydrogen peroxide in aqueous media. Experiments were carried out for several types of composites (PET TeMs with different pores density, different time of silver deposition) as well as for different temperature regimes.

Experiment's details

Preparation of Ag/PET TeMs composite

PET film (Hostaphan® RNK, Mitsubishi Polyester Film, nominal thickness 12 μm) was irradiated with Kr ions (energy: 1.75 MeV per nucleon; the preferred ion fluencies were 1×10^9 and 4×10^7 per cm^2) at the DC-60 cyclotron in Astana and etched in NaOH solution (2.2 M at 85 $^\circ\text{C}$), with finally cylindrical pore sizes as 100 ± 5 and 430 ± 20 nm respectively). The effective pore sizes of parent template as well as the inner diameter of embedded NTs were estimated by gas (compressed air was applied) permeability technique using Hagen–Poiseuille equation.

The improved electroless deposition technique reported by F.Muench [1] was used for Ag/PET preparation and plating solutions containing AgNO_3 (17 mM), potassium sodium tartrate (120 mM) and pyridine (50 mM) as a complexing agent was applied.

In all experiments deposition took place at 4 $^\circ\text{C}$ within desired time (30, 60, 180 and 300 min) afterward Ag/PET composite membranes were washed in water and ethanol and dried with air.

Evaluation of catalytic activity

Generally the catalytic decomposition of H_2O_2 was carried out in a 250 mL round-bottomed flask immersed in a water bath. For each test, 5.75×4.25 cm of the Ag/PET catalyst was used.

The reaction time was recorded after the addition of 50 mL (1.02 wt% H_2O_2) solution preheated at the same operating temperature. Standard H_2O_2 solution was prepared by dilution and its concentrations was standardized by standard potassium permanganate solution. A graduated glass burette was used to measure the volume of O_2 . After complete decomposition of H_2O_2 , the catalysts were thoroughly rinsed with deionized water.

Influence of Ag/PET TeMs deposition time on catalytic degradation of H_2O_2

Here we are present results on influence of deposition time of silver inside the pores of PET TeMs on catalytic ability of these composites. Thus, the deposition was carried out within 30, 60, 180 and 300 minutes. By low-temperature electroless deposition technique we were able to prepare a series of PET TeMs with embedded gold and silver nanotubes. Figure 1, shows SEM surface images of synthesized Ag/PET composites as well as cross-sectional view of silver NTs arrays after template dissolving.

As it is seen, the obtained nanostructures have a tubular morphology and are uniform in terms of their length (12 μm). The inner diameter of the silver nanotube walls was also determined. As it could be seen from table 1

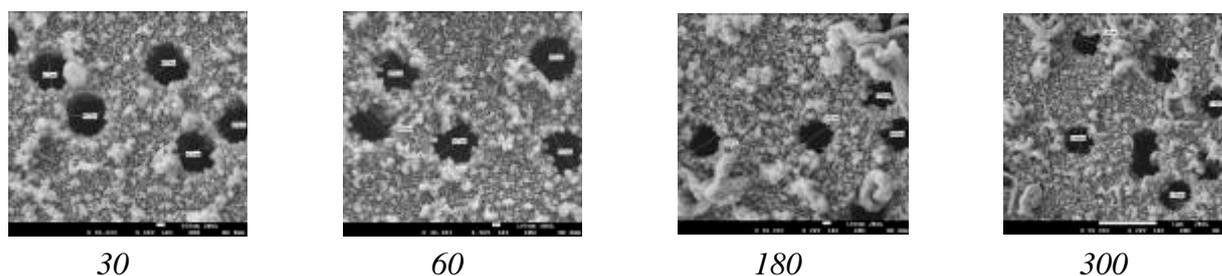


Fig.1 A closer surface view of Ag/PET composites with different deposition time and SEM images of template-free silver NTs arrays electrolessly deposited on PET TeMs.

Table – 1. Diameters of silver NTs deposited in PET TeMs for 4×10^7 template

Deposition time, min	30	60	180	300
Inner diameter of Ag nanotubes, nm	486.7 ± 6.7	470.4 ± 4.4	357.4 ± 5.6	348.8 ± 10
Wall thickness, nm	40.45 ± 0.8	48.6 ± 1.5	105.1 ± 2.9	109.4 ± 4.3

The kinetics of the catalytic decomposition of hydrogen peroxide was conducted at 25°C for all deposition times. The analysis of the experimental data has been carried out on the assumption that the decomposition of H_2O_2 is a first order process. The volume of the evolved oxygen was plotted as a function of time (Fig. 2).

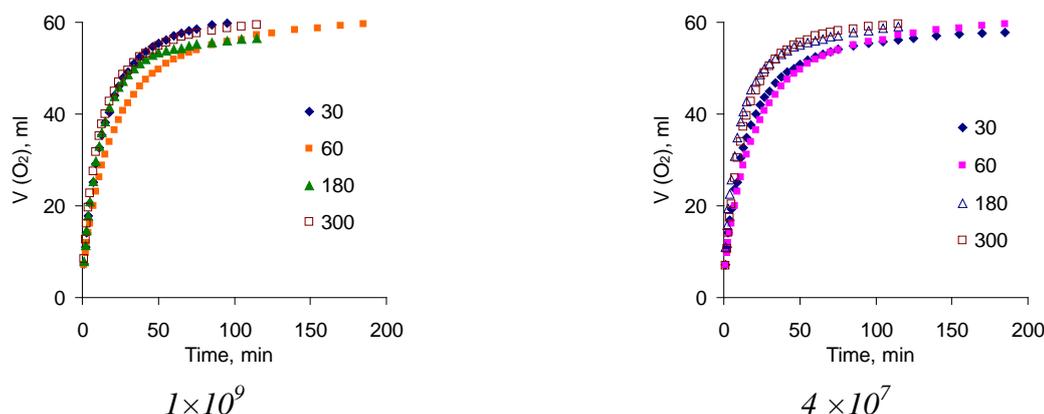


Fig. 2 Catalytic decomposition of H_2O_2 over Ag/PET composite: evolving of oxygen as a function of time for different pore size template .

From the slope of the straight lines the apparent rate constants k as well as reaction order were obtained (Table 2).

Table 2 Kinetic data of decomposition of H_2O_2 on the surface of Ag/PET samples with different pore density.

Deposi-	Ag/PET composite
---------	------------------

tion time, min	1×10^9					4×10^7				
	N	Total reaction time τ , min	V (O ₂), ml	k(*10 ⁻³) min ⁻¹	$\bar{k} \pm \delta$	N	Total reaction time τ , min	V (O ₂), ml	k(*10 ⁻³) min ⁻¹	$\bar{k} \pm \delta$
30	0,92	185	60,3	9,8	$8,7 \pm 0,95$	0,90	170	60,1	14	$12,3 \pm 1,49$
	0,97	220	58,6	8,1		0,92	185	57,8	11,7	
	0,94	240	61,2	8,2		0,94	200	58	11,2	
60	0,95	185	52,8	15,18	$15,2 \pm 6,4$	0,95	185	58,62	13	13 ± 3
	0,93	155	60,7	16,1		0,96	185	60	11,6	
	0,92	155	58,4	16,4		0,95	170	58,4	12,2	
180	0,92	240	62,8	9,3	$11,1 \pm 1,85$	0,92	170	60	12,5	$12,4 \pm 0,38$
	0,98	170	58,2	11	\pm	0,93	170	59,2	12,6	
	0,92	170	59,9	13	$1,85$	0,92	185	59,4	12	
300	0,97	240	61,8	8,4	$9,33 \pm 0,83$	0,92	140	59,9	17	$14,9 \pm 2,15$
	0,95	200	61,2	10	\pm	0,95	140	60,4	15	
	0,94	220	62	9,6	$0,83$	0,94	185	60	12,7	

As seen from the data presented in the Table 2, for the samples of Ag/PET composite deposited in a matrix with the density of pores 1×10^9 , the most efficient deposition time - 60 minutes. It could be explained by the several reasons i.e. by increasing deposition time, the formation of large agglomerates of silver on the membrane surface and clogging of membrane channels was observed, and in this case area inside the channels became less active for catalyzing.

Constant rate of Ag/PET composites prepared from 4×10^7 PET TeMs increases linearly with increasing deposition time. However, increasing the deposition time from 30 to 300 minutes, and the constant rate is increased by only 16.78%.

Thus for further experiments on influence temperature on the catalytic capacity of Ag/PET composites the most effective deposition time was chosen.

Effect of operating temperature on the catalytic degradation of H₂O₂ in presence of Ag/PET catalyst

The influence of reaction temperature was studied by checking the performance of the Ag/PET catalyst at temperatures in the range 25-45 °C. It was observed that an increase in reaction temperature is paralleled by an increase in the decomposition rate of hydrogen peroxide (Tables 3-4).

Table 3. Kinetic data of decomposition of H₂O₂ on the surface of Ag/PET samples with 4×10^7 l/ion pore density.

T, °C	N	Total reaction time τ , min	V (O ₂), ml	k(*10 ⁻³) min ⁻¹	$\bar{k} \pm \delta$
25	0,96	185	60	0,0116	$13,02 \pm 3,05$
	0,95	240	57,8	0,0094	
	0,95	170	58,4	0,0122	
	0,93	140	58,1	0,0174	
	0,92	155	58,8	0,0145	
30	0,98	260	67,4	0,0081	$8,77 \pm 0,8$
	0,94	240	60,2	0,0085	
	0,93	220	64,7	0,0097	
35	0,95	185	66,4	0,0110	$11,67 \pm 0,58$
	0,95	170	64,5	0,0120	
	0,95	185	70,4	0,0120	

40	0,96	105	69,6	0,0201	20,7 ± 4,03
	0,93	85	66	0,0250	
	0,95	125	70,2	0,0170	
45	0,96	70	77,4	0,0329	29,9 ± 2,61
	0,92	75	74,8	0,0289	
	0,96	85	77	0,0280	

As can be seen from the data presented in Tables 3-4, the rate constant increases linearly with increasing temperature.

However, at temperatures above 40 degrees, partial destruction of the catalyst and it is not possible to reuse. The most satisfying to our requirements results were obtained for the reaction at 25 degrees, i.e., at room temperature. These conditions allow carrying out the reaction without any additional heating equipment and can be easily transposed into industrial scales.

By testing the catalytic activity at different temperatures the apparent activation energy (E_A) was calculated via the Arrhenius plot of the $k_{app}=f(1/T)$ (Fig.4)

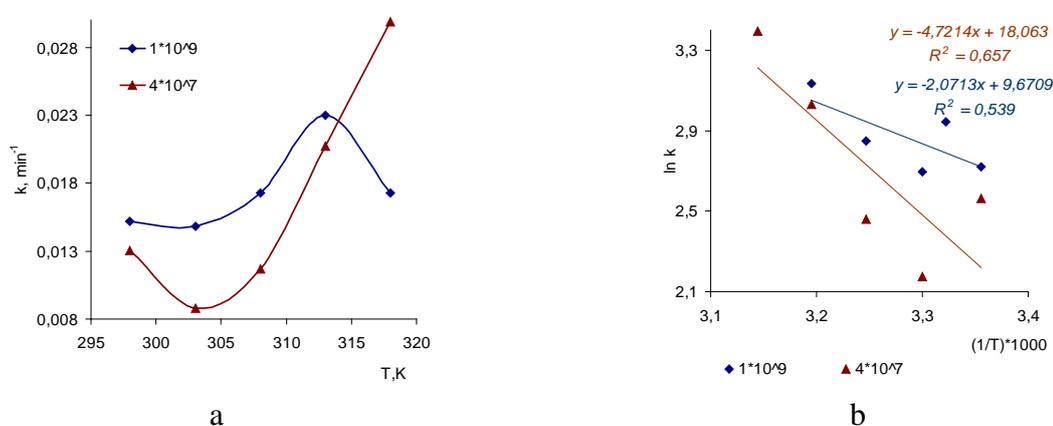


Fig.4 Effect of temperature on decomposition of hydrogen peroxide by composites based on PET TeMs with embedded silver NTs(a) and Arrhenius plot for studied reaction (b).

An activation energy was found to be $E_A = 17.22 \text{ kJ mol}^{-1}$ and $E_A = 39.25 \text{ kJ mol}^{-1}$ for Ag/PET composites prepared from 1×10^9 and 4×10^7 PET TeMs respectively.

The reusability of the catalyst is an important factor from economic and environmental point of views and has attracted much attention in recent years. The reusability of prepared Ag/PET composites (pore density 1×10^9) was checked consecutively for three times at 40°C (Table 5)

For decomposition of hydrogen peroxide reaction, the reaction rate of second cycle is decreased in 1.4 times

Table 4. Kinetic data of decomposition of H_2O_2 on the surface of Ag/PET samples with 1×10^9 1/ion pore density.

T, °C	N	Total reaction time τ , min	V (O_2), ml	$k (*10^{-3}) \text{ min}^{-1}$	$\bar{k} \pm \delta$
25	0,98	280	53,2	0,0085	15,183 ± 6,369
	0,98	325	49	0,0072	
	0,97	95	48,8	0,0190	
	0,98	95	47,2	0,0240	
30	0,93	155	60,7	0,0160	14,833 ± 5,037
	0,92	155	58,4	0,0164	
	0,96	155	51,2	0,014	
	0,98	140	57,4	0,017	
	0,92	155	55,4	0,012	

	0,93	125	60,8	0,024	
35	0,98	115	60,4	0,020	17,25 ± 2,217
	0,99	140	59,2	0,015	
	0,99	140	58,3	0,016	
	0,98	140	60,8	0,018	
	0,99	115	62,5	0,017	
40	0,99	155	62	0,011	23,0 ± 13,7
	0,98	220	64,4	0,010	
	0,99	105	53,8	0,020	
	0,95	55	50,6	0,040	
	0,96	50	55,6	0,040	
	0,98	155	77,2	0,017	
45	0,98	125	71,4	0,018	17,25 ± 0,957
	0,97	125	74,4	0,018	
	0,97	105	73,2	0,016	
	0,98	155	77,2	0,017	
	0,98	125	71,4	0,018	
	0,98	125	71,4	0,018	

Table 5 Reusability of the Ag/PET catalyzt (1×10^9)

Cycle	N	$k(*10^{-3}) \text{ min}^{-1}$	Conversion of initial reagent, %	V (O ₂), ml
1	0,94	14,6	74.50	59,6
2	0,92	10,3	75.63	60,5
3	0,92	9,4	76.75	61,4

Here we demonstrated detailed results on application of Ag/PET TeMs composites for catalytic degradation of hydrogen peroxide. At the first time the influence of template pore density on catalytic ability of Ag-based composites was demonstrated for samples with different deposition time. The most effective results were observed for 60 minutes deposited samples. Also dependence of operating temperature on constant rate was studied.

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ПОЛУЧЕНИЕ ХРОМСОДЕРЖАЩИХ СОЕДИНЕНИЙ ИЗ ОТХОДОВ ХРОМИРОВАНИЯ ГАЛЬВАНИЧЕСКИХ ПРОИЗВОДСТВ

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Гальванические покрытия стальных изделий хромом широко используется в технике. Хромирование используется в декоративных целях, для снижения трения, повышения износостойкости изделий и коррозионной стойкости. Вместе с тем, гальваническое