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Scientific Research of the Influence of the Vacuum Metalling Over the Operational Characteristics of the Ultrafiltering Polymer Membranes

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ABSTRACT *The transport and selective characteristics of the vacuum metalling ultrafiltering polymer membranes are investigated. Magnetronic spread of the iron – chrome – nickel alloy in made and is traced the influence of the time of exposition of the same alloy over the characteristics of the membranes. The influence of the additional metal – oxide sub-layer is read, in the form of intermediate sub-layer.*

KEYWORDS: polymer membranes, ultrafiltration, magnetronic spread, iron-chrome-nickel alloy.

Introduction

The increase in production of metallic polymers and widening in the sphere of their application are prompted by the possibility and the methods of vacuum metalling to obtain materials that combine the good operating capacities of metals and polymer's surface and the parameter's regime when applying the layer (Ulbricht,M , 2006).

The vacuum metalling of the membranes has the purpose to change the transport and selective characteristics of the membranes and make them applicable for the solution of concrete optimizing problem (Baker, R.W. 2004).

On the other hand, the different regimes of vacuum metalling(a temporary exposition) brig for the setting up of a metal layer over the polymer's surface of the membrane, and that layer appears to be defining for the characteristics of the membranes, and it plays the role of a second selective layer of the already formed membrane (Sacher,E.,2002).

Experimental

One method for the preparation of polymer membranes is the phase inversion (Kim et al.,2002; Kobayashi et al., 1997). The membranes are formed in laboratory conditions through a phase-inversion method, more popular as the methods of Loeb (Loeb S., et al. 1962). The obtained membranes have the following composition PAN – 16.25 mass%, PMMA – 0.25 mass%, $CuSO_4.5H_2O$ – 1.50 mass %. The addition of PMMA in the polymer solution in the process of phase – inversion helps for the setting up of membranes with greater porousness and productivity. The change in the transport characteristics of these membranes is due to the incompatibility of the two polymers. In order to activate the polymer's surface and to increase the adhesion connection between the polymer membrane and the metal cover is made a preliminary modification with alkaline and salt-acid solutions of $SnCl_2.2H_2O$(Lipin, Yu.et al.1977).

The metal covers are obtained in a vacuum installation BUP-5 (Russia), by spreading iron – chrome – nickel alloy. Magnetron spread is done of the alloy target with a starting vacuum in the chamber $P_H=1.10^{-3}$ Pa working gas medium Ar with purity 99,99% under the pressure of $P_p=4.10^{-2}$ Pa and a specific power of spreading $N_p=5,7$ W/ cm^2. The time of precipitation of the cover is 5,10,15,20 and 25 s. The power of the continuous current source feeder of the electric current from 0 A up to 10 A.

In order to define the working characteristics of the metal membranes is used a laboratory module " Sartorius" type SM – 165 (England). To define the selectivity of the membrane against the calibrator is defined with the help of UV/ VIS spectrophotometer " Unican - 8625" (France) with the length's wave 280 nm.

Results and Discussion

To obtain membranes from polymer mixtures with the participation of different salt (acids) in practice means that it goes with a maximum homogeneous distribution of the polymer in the solvent, and is characterized with a small speed of the phase inversion, and the setting up of a structure with reduced porousness. The obtained membranes possess the highest density on the surface layer, which causes new conditions of vacuum metalling (Charbonnier, M. et al.(2003). Following the basic characteristics of the membranes is not noticed the tendency of lowering (reducing) the waterpermeability with the increase of the time of the exposition in direct metalling , fig.1, and an increased waterpermeability when these is metalling with metal-oxide sub-layer, fig.1b. Naturally, the mechanism of the process can be registered and explained is structures with a minimal porous size. If there is some dependency between the presence of the oxide sub-layer and its position and influence over the next metalling cover most probably it will be read smallest radius porousness.

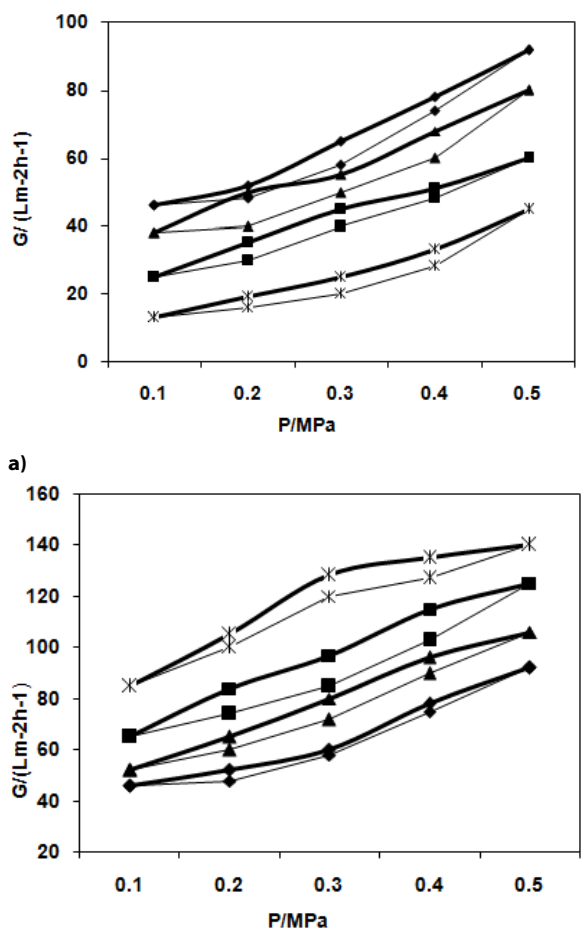
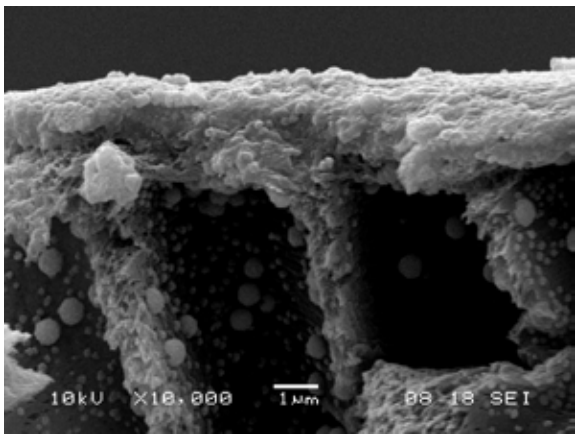


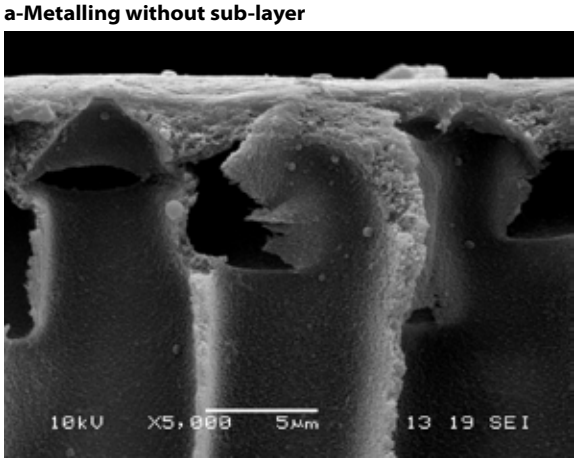
Fig.1. Water permeation flux of the membranes, first modified with 50g/l $SnCl_2.2H_2O$ and 100g/l HCl ,vacuum metalling with chrome-nickel alloy:a- without sub-layer, b-with oxide sub-layer. ▲-5 s, .▲-15 s, ж-25 s,▲-pure membrane.

When the oxide sub-layer is absent fig.2, the metalling atoms enter the

porous structure and congest the diffusion layer.
When the time of exposure increase the density of that layer increase, and it lowers the waterpermeability of the membrane. When there is oxide layer, the zone around the maximum big porous structure (fig.2b) appear to be concoction of precipitated oxide atoms.



a-Metalling without sub-layer



b-Metalling with sub-layer
Fig.2 SEM of membranes, first modified with 50g/l $SnCl_2.2H_2O$ and 100g/l HCl ,vacuum metalling with chrome-nickel alloy: a - without sub-layer, b-with oxide sub-layer.

In that way they (the oxide atoms) build a barrier layer, which does not allow the atoms of the iron, nikel and chrome to enter the porous structure, and to block it. In this way, the structure of the membrane in the selective zone (fig.2b) is characterized with the presence of the metalling layer without its registering in the porous structure. In practice, the usage of metal-oxide cover favours the setting up of two consecutively situated layers which define the positively changing characteristics of the membranes.

The setting up of a similar structure of the membranes should ensure an increased selectivity towards rating Albumin calibrators. Wheter this selectivity depends on the exposition of the metalling cover, we can read through the results from fig.3

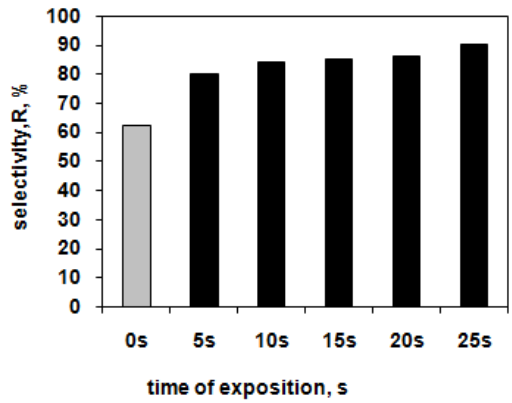


Fig.3. Selectivity towards Albumin (under 0,3 MPa) of polymer membranes, first modified with alkaline solution ,vacuum metalling with chrome-nikel alloy.

The range of the vacuum metalling modification can be divided into two periods. The first essential change appears with the first treatment of 5 s. The time is enough to make a cover which increases selectivity of the membrane from 60 to 80%. We can suggest, that in practice a complete metal cover is not built. But the precipitation of the metal ions is done in the zones where an oxide pad is stored i.e. in practice it means that selectively are blocked the macropores of the membrane. This can explain the sudden increase in the selectivity at exposition of 5 s. In the interval of 5s to 25s the complete building of the metalling cover and its selectivity increase with more than 10%.

The increase in selectivity of the membranes with 30% though one selective modification show that the chosen condition do not influence negatively the porous structure and its stability in the period of modification.

Conclusions:

1. The possibility of vacuum metalling of polymer membranes with different membrane surface is proved in the regime of direct metalling and with the usage of preliminary modification of an oxide sub-layer.
2. Through a direct vacuum metalling, with the increase of time exposition from 5s to 25s is reduced the waterpermeability, and the selectivities of the membranes increase insignificantly.
3. When metal-oxide sub-layer is used, the vacuum metalling leads to an increased waterpermeability for membranes which consist of 16,25 mass %PAN, 0,25 mass % PMMA, 1,50 mass % $CuSO_4.5H_2O$ and it reaches up to 140 L $m^{-2} h^{-1}$.
4. Through the method of SEM analysis is proved the mechanism of precipitation of the oxide and metalling layer which explains the increasing permeability through the membranes.

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