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Trembus I.V.
National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute"

Mykhailenko N.V.
National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute"

Hondovska A.S.
National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute"

MEMBRANES BASED ON MODIFIED CELLULOSE FIBERS. A REVIEW

In the article it was analyzed main oxidation-organosolv methods of obtaining cellulose from plant raw materials. The expediency of usage peroxyacids as delignifying reagent was described and advantages compared to traditional methods for cellulose obtaining were analyzed. Methods of modifying cellulose fibers for obtaining materials with specified adsorption properties in relation to dyes and heavy metal ions as well as for air purification were described. The influence of solution pH, duration and temperature of process as well as the initial solution concentration on the cellulose membranes efficiency were analyzed. The main characteristics of membranes and conditions for their usage were highlighted. It is shown that various modified fibers can be used for selective adsorption of individual impurities and show ability for complex removal. Methods of removing crystal violet, methyl blue from aqueous solutions, anionic dyes adsorption and various organic liquids using environmentally friendly cellulose membranes were described. The possibility of removing Cu^{2+}, Cd^{2+}, Pb^{2+} and As^{5+} ions with the assistance of modified cellulose material was considered. Such cellulose material has higher adsorption capacity in case of Cu^{2+}, Cd^{2+}, and Pb^{2+} ions removal. Adsorbents obtained by cellulose fibers treatment with concentrated sulfuric acid followed by chemical surface modification with N-aminoguanidine functional groups show high selectivity for precious metals adsorption from hydrochloric acid medium. It was shown that filter material obtained from carboxylated cellulose has excellent adsorption capacity in relation to Pb^{2+} and methylene blue from aqueous solutions. Membranes based on modified cellulose acetate nanofibers can be used as effective material for Hg^{2+}, Cu^{2+}, and Cd^{2+} adsorption. Such material has much higher adsorption capacity for Hg^{2+} than for Cu^{2+} and Cd^{2+}. Positive influence of mercerization process for cellulose filter materials production was shown. It was determined that main advantages of mentioned cellulose filter materials are high adsorption capacity, possibility of usage in various conditions and ability to be regenerated and reused.

Key words: *membrane, organosolvent cellulose, modified cellulose fibers, membranes, adsorption, dyes, heavy metal ions.*

Formulation of the problem. Environmental pollution control is one of the main scientific research topics today. There are more and more environmental problems caused by society economic development especially those related to water purification [1].

It is considered that cellulose is the most common renewable polymer in nature and also it fairly cheap and promising raw material for obtaining various functional products [2].

In addition, surface of cellulose is sensitive to chemical modifications due to hydroxyl groups which presence in its structure and can react with primary and secondary alcohols. One of cellulose modification goals is to obtain materials with greater adsorption capacity for pollutants which are present in aqueous environment compared to original biopolymer [3, 4].

Analysis of recent publications and research The adsorption process is widely used for treatment of wastewater which contains toxic dyes due to its high efficiency, simplicity and low cost. A lot of attention is paid to cellulose as a polymer which can be used for synthesis of new filter material [5].

Constant growing of costs for filter materials and increasing of environmental requirements have led to significant amount of research work aimed at developing technologies for obtaining new inexpensive adsorbents from renewable resources. First of all, the advantage of usage cellulose as basis for new filter material is its prevalence, relatively low cost, modern methods of it obtaining, namely environmentally friendly oxidation-organosolv methods and relative easiness to chemical modification [6].

Setting objectives. The purpose of the work is to consider modern environmentally friendly methods for obtaining cellulose, main modification methods and to determine possibility of usage such membranes for removing impurities from aqueous solutions and purify the air.

Presentation of the main research material. The search of new waste-free technologies for cellulose obtaining is conducted in various spheres including cellulose biological synthesis, enzymatic wood delignification and solvolytic plant materials delignification in organic solvents [7, 8].

Highly selective reagents such as peroxoformic acid or peroxoacetic acid are successfully used for obtaining cellulose from wood and non-wood plant materials, especially, agricultural waste.

Solvent delignification methods make possible to utilize the hemicelluloses from plant raw materials and isolate reactive lignin which can be easily converted into various valuable chemical products [9, 10]. Such processes are characterized by significantly shorter duration if compare with the traditional sulfate and sulfite delignification methods. Organic solvents can be regenerated by distillation with little energy consumption. Recycling of organic solvents reduces the solutions volumes which spent in processing. As a result, it reduces overall capital costs and reduce industrial effluents which makes such technologies environmentally friendly and economically attractive.

Development directions in technology of organosolv plant raw materials processing are related to usage oxidants which contributes to cellulose content increasing in obtained product [11].

Hydrogen peroxide and ozone are most often used as oxidants which are considered to be environmentally friendly and capable to delignify plant biomass. The process is carried out in solutions of organic acids or alcohols [11-13].

Usage of oxidative-organosolv methods for cellulose production allows to obtaining products with high quality, yield and low residual lignin content. Functional characteristics of this material allow use it as alternative raw material for modification with various substances [14].

Synthetic dyes are considered to be environmentally dangerous due to their high toxicity, color and inability to decompose, but despite of this, textile, printing and plastic industries are widely used them. As a result, solving issue of wastewater pollution with dyes has become relevant today.

Modified cellulose materials are promising adsorbents because they contain large number of functional groups which capable to retain dye molecules. A lot of modification methods which give necessary adsorption properties to cellulose for effective dye removal are already known [15].

Cellulose modification with sulfosalicylic acid and glycidyl methacrylate effectively increases its electronegativity which leads to increasing adsorption capacity for crystal violet removal from aqueous solutions. Solution pH, duration, temperature and initial solution concentration have directly effect to the efficiency of such membrane. Great advantage of this adsorption material is its ability to be regenerated and possibility to be repeated use [16].

Material based on dialdehyde nanocellulose and amphoteric polyvinylamine is excellent adsorbent for anionic dyes removal. It is demonstrates maximum removal efficiency in acidic conditions due to amino groups protonation. Such filter material can be used for removal various types of anionic dyes and it has highest adsorption capacity to the congo red [17].

Graphene oxide airgel has great interest for wastewater treatment due to its porous structure and high specific surface area. Carboxymethyl cellulose in combination with graphene oxide airgel can be obtained using modified hydrothermal reaction of ethylenediamine reduction. This material is hydrophilic, stable and demonstrates excellent adsorption capacity for organic liquids. Such membrane can be used more than one time after regeneration. The filter material based on carboxymethyl cellulose and graphene oxide is able to effective adsorbtion of rhodamine B dye in aqueous solution and, as a result, it can be potentially used for wastewater treatment [18].

Environmentally friendly membranes which are made with polyvinyl alcohol and carboxymethyl cellulose with graphene oxide are used for improved removal of methylene blue from water solutions. Bentonite addition to membranes significantly improves adsorption properties of material in relation to dyes. To the adsorption capacity of filter material in relation to methylene blue is significantly influence filter solution pH, initial dye`s concentration, contact duration with membrane and solution temperature. So, this kind of filters are stable, effective and can be used in practice as a reusable adsorbent for anionic dyes in wastewater treatment [19].

Environmentally friendly membranes based on polyvinyl alcohol, carboxymethyl cellulose and zeolite are very suitable for adsorption of methylene blue from aqueous solutions. This filter material shows uniform morphology, sufficient mechanical strength and good thermal stability. Adsorption efficiency increases with more zeolite content in the membrane, contact duration with solution and high initial dye concentration [20].

Filter material for dyes removal from aqueous solutions can be obtained by cellulose modification with maleic anhydride and diatomite which is pretreated with alkali solution. To increase size of the pores in filter material $CaCO_3$ solution is added during cellulose modification and, as a result, it improves adsorption properties. Adsorption capacity for ordinary filter paper and alkali-treated diatomite is extremely low. In the same time membranes made from cellulose and diatomite already have higher adsorption capacity compared with primary materials. The adsorption capacity in relation to basic dyes such as methylene blue and methyl violet is doubled after cellulose modification with maleic anhydride. In this case dyes adsorption depends on various factors such as mixing duration, temperature and the most important factor is pH medium. The efficiency of dye removal from aqueous solutions reaches maximum in an alkaline medium at $pH > 7$. In this case adsorbent has negative charge and, as a result, it increases its affinity for cationic dyes [21].

Usage of methyl-3-methoxysilane for chemical cellulose modification makes possible to obtain hydrophobic cellulose filter for air purification which has sufficiently developed porous surface structure which is obtained by freeze-drying. As a result, such filter is able to work effectively under conditions of high air humidity. Addition of methyl-3-methoxysilane to cellulose fibers improves structure of filter pores, increases specific surface area and porosity which in turn has positive effect on the filtering solid particles efficiency. In addition, the low pressure drop in filter effectively improves air purification quality. At the dew point temperature filter can effectively work despite of increasing in filtration resistance. It should be noted that resistance rate in such membranes is much lower than that of hydrophilic filter material. In addition, hydrophobic modification can effectively improve water vapor transmission rate through filter. This indicates that hydrophobic modification filter has excellent water resistance and, at the same, time air permeability. Prepared hydrophobic filter can be used in indoor air purification, breathing masks, cars and exhaust gas treatment. Long-term and stable filtration in environment with high humidity testifies to effectiveness and stability of this membrane [22].

Such metals ions like Cu^{2+}, Cd^{2+} and Pb^{2+} have rather high toxic hazard that attracts attention due to their tendency to accumulate in humans and animals. These compounds do not break down into harmless end products in metabolism process and accumulate in the body and, as a result, create great danger for organisms [23].

Cellulose filter material containing independent methylbenzaniline groups have good removal ability for Pb^{2+} and Cu^{2+} ions from water and beside this high antimicrobial activity. This material has great ability to adsorb in wide range of pH values both in acidic and alkaline medium. Filtering capacity of such material increases as pH value is grown to weakly acidic and decreases with further growth. This trend is explained by the fact that in high H^+ ions concentration there is competition between them with metal ions for coordination with active azomethine groups and, as a result, percentage of metal ions removal decreases. It should be noted that such material is quite effective even in strongly acidic environment which makes it possible to use such membranes in industry [24].

Cellulose is modified by treating with aqueous NaOH solution to increase reactivity which causes changes in crystalline structure. During mercerization alkali penetrates cellulose fiber and causes rearrangement of crystalline structure and, as a result, it swells. Polysaccharide chains change their structure during the process and fibers disorder increases. These changes lead to fact that specific surface of fibers increases and hydroxyl groups in cellulose structure become more accessible and reactive. These changes are irreversible and usually accompanied with crystallinity decrease and polymerization degree due to glycosidic bonds hydrolysis [25, 26].

During cellulose filter materials production mercerization has rather positive effect on final product quality because separation of polysaccharide chains is increases. In that way, it simplifies process of succinic anhydride penetration which gives to cellulose good adsorption properties. Modified mercerized cellulose shows higher adsorption capacity during removal of Cu^{2+}, Cd^{2+} and Pb^{2+} ions from aqueous solutions if to compare with modified but not mercerized cellulose. It was established that efficiency of metal ions adsorption directly depends on carboxyl groups number introduced to cellulose [27].

Water pollution with As^{5+} is considered to have serious and global threat to humans due to its high toxicity. Cellulose modified with 3-mercapto-propanoic acid can act as adsorbent to remove arsenate from aqueous solutions using batch membrane distillation process. Important step for covalent attachment of 3-mercaptopropanoic acid to cellulose filter membrane surface is primary acid activation with dicyclocarbodiimide. During this process dicyclocarbodiimide reacts with 3-mercapto-propanoic acid to intermediate form O-acylisourea which has high reactivity. It is advisable to carry out mercerization of cellulose material with aqueous solution of NaOH for nucleophilicity increasing of hydroxyl groups [28].

Adsorption cellulose filter material capacity after modification in relation with As^{5+} ions increases significantly. Thiol groups have important role in adsorption process. As a result, if cellulose substitution degree is greater, it will have better adsorption. In addition, the cellulose membrane with 3-mercapto-propanoic acid shows good stability and performance after regeneration which makes possible it practical usage. In conclusion, such unique modified cellulose membrane with 3-mercapto-propanoic acid effectively removes arsenate by filtration and can be simple and effective way for removing arsenic from drinking water [28].

Filter materials obtained by treating cellulose fiber with epichlorohydrin or concentrated sulfuric acid and subsequent surface chemical modification with N-aminoguanidine functional groups show high adsorption capacity to Au^{3+}. Such adsorbents show phenomenal selectivity for precious metals removal from hydrochloric acid medium. It should be noted that base metal ions are not adsorbed at all under such conditions. It is possible due to the fact that Au^{3+}, Pd^{2+} and Pt^{4+} form chloride anions in hydrochloric acidic conditions and adsorption of these ions occurred due to ions pair formation on positively charged nitrogen atoms of adsorbent. Therefore, such membranes can be very promising for selective gold extraction from solutions with significant content of other metals [29].

Carboxyl cellulose oxidized with 2,2,6,6-tetramethylpiperidine-1-oxyl demonstrates universal capacity to dyes and heavy metal ions removal. The morphology of surface and cross-section show that cellulose oxidized in this way has micro- and sub-microstructures in its composition that increase functionality and water permeability of filter material. As a result, carboxylated cellulose material has excellent adsorption capacity for Pb^{2+} and methylene blue from aqueous solutions. The actual adsorption capacity of carboxylated cellulose fabrics for Pb^{2+} and methylene blue is close or even higher according to results of most adsorbents. Adsorption mechanism shows that adsorption of Pb^{2+} and methylene blue occurs not only on surface but also in the internal porous structure of carboxylated cellulose fibers which has positive effect on the filtration process. If compare with unmodified cellulose such membranes are more effective in filtering multi-component wastewater. In addition, durability of fabric filters made with carboxylated cellulose is confirmed by repeated use [30].

Cellulose acetate nanofibers which produced by electrostatic spinning technology have high porosity, large specific surface area, high permeability, small fiber diameter and can be used for filtering small diameter particles. Thus, such fibers are suitable for filter materials production [31]. Number of functional groups increases due to high specific surface area on cellulose acetate material and, as a result, it improves heavy metal ions adsorption and separation in fibers [32].

Modified cellulose acetate nanofiber made with methacrylic acid by electrostatic spinning technology can be used as effective material for Hg^{2+}, Cu^{2+} and Cd^{2+} adsorption. Such material has much higher adsorption in relation to Hg^{2+} than Cu^{2+} and Cd^{2+}. During ions removal process it was noticed that adsorption capacity to Cu^{2+} and Cd^{2+} decreases slightly but remains unchanged in relation to Hg^{2+} [33].

Membrane made with mixture of lignin and cellulose acetate which produced by electrostatic spinning technology has ability to remove Ag^{+} and Cu^{2+} from aqueous solutions. It is shown that the adsorption capacity to Cu^{2+} is greater than that to Ag^{+}. Removal efficiency improves with increasing temperature and initial concentration of metal ions. The adsorption capacity of such material largely depends on solution pH and shows best efficiency in weakly acidic medium. It is shown that the mass ratio of lignin and cellulose acetate has little effect on adsorption capacity [34].

Conclusions. Modified cellulose fibers which made by environmentally friendly method using oxidative-organosolv delignification is alternative basis for filter membranes. Such filter material does not require usage toxic compounds in it production which makes it quite promising in modern conditions. That is why it can be successfully used for removal dyes from industrial wastewater, metal ions from aqueous solutions and air purification. Various modifications of this material show selective adsorption capacity for various impurities. Thus they can be used for removal of specific substances and have complex action for different chemical compounds. The main advantages of such filter materials is high regeneration capacity and, as a result, possibility to be repeated use without reducing its efficiency. Such cellulose membranes properties make possibility to use them in various human activity spheres and make it possible to reduce harmful effects of toxic substances on humans.

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Трембус І.В., Михайленко Н.В., Гондовська А.С. МЕМБРАНИ НА ОСНОВІ МОДИФІКОВАНИХ ВОЛОКОН ЦЕЛЮЛОЗИ. Огляд

У статті проаналізовано основні оксисно-органосольвентні способи одержання целюлози із рослинної сировини. Обгрунтовано доцільність використання пероксокислот в якості делігніфікуючого реагенту та проаналізовано їх переваги у порівнянні з традиційними способами варіння целюлози. Описано методи модифікації целюлозних волокон для отримання матеріалу із заданими властивостями адсорбції по відношенню до барвників та іонів важких металів, а також для очищення повітря. Проаналізовано вплив pH розчину, тривалості і температури процесу, а також початкової концентрації розчину на ефективність роботи целюлозних мембран. Висвітлено основні характеристики мембран та умови для їх застосування. Показано, що різні модифіковані волокна можуть використовуватися для вибіркової адсорбції окремих домішок, так і проявляти здатність до комплексного видалення. Описано способи видалення кристалічного фіолетового, метилового синього з водних розчинів, адсорбції аніонних барвників та різних органічних рідин за допомогою екологічно чистих целюлозних мембран. Розглянуто можливість видалення іонів Cu^{2+}, Cd^{2+}, Pb^{2} і As^{5+} за допомогою модифікованого целюлозного матеріалу. Показано позитивний вплив процесу мерсеризації у виробництві целюлозних фільтрувальних матеріалів. Така целюлоза має на порядок вищу адсорбційну здатність у випадку видалення іонів Cu2+, Cd2+та Pb2. Адсорбенти отримані обробкою целюлозного волокна концентрованою сірчаною кислотою з подальшою хімічною модифікацією поверхні функціональними групами N-аміногуанідину показують високу селективність до адсорбції дорогоцінних металів із середовища соляної кислоти. Показано, що фільтрувальний матеріал з карбоксильованої целюлози має чудову адсорбційну здатність по відношенню до Pb2+ і метиленового блакитного із водних розчинів. Мембрани на основі модифікованого нановолокна з ацетату целюлози можуть використовуватися як ефективний матеріал для адсорбції Hg2+ , Cu2+ та Cd2+. Такий матеріал має набагато вищу адсорбцію Hg2+, ніж Cu2+ і Cd2+. Було встановлено, що основними перевагами названих фільтрувальних целюлозних матеріалів можна назвати високу адсорбційну здатність, можливість використання у різних умовах та здатність до регенерації та повторного використання.

Ключові слова: *мембрана, органосольвентна целюлоза, модифіковані целюлозні волокна, мембрани, адсорбція, барвники, іони важких металів.*