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Application of composite materials based on polyvinyl alcohol in phytoremediation soil

Oil production, transportation, transshipment and storage lead to accidental soil pollution with oil products. To solve this problem, the researchers proposed the possibility of using polymer films for the rehabilitation of oil-contaminated soils. In the present work, in order to clean the soil from oil products, films based on polyvinyl alcohol and chitosan were obtained, their main physical and mechanical properties were investigated. The results of IR spectrometric analysis showed good compatibility of the starting components. It was found that the films obtained possess rather high indices of mechanical strength. Moreover, films containing 80 % synthetic polymer — PVA showed the highest mechanical properties. The latter is apparently associated with the formation of a network of intermolecular hydrogen bonds between chitosan and PVA, which favorably affects their operational characteristics. Pollution modeling was carried out by applying oil to the soil of the Karazhanbass field. The resulting films based on polyvinyl alcohol and chitosan were used as recultivates, which were applied in concentrations of 0.2 g/kg of soil. The effectiveness of remediation of oil-contaminated soils was evaluated by measuring the content of oil products and conducting phytotests of the studied soil samples. The results of the assessment of the degradation of oil products in the process of reclamation and the phytotesting indicators obtained in the dynamics of the experiment allow us to conclude the effectiveness and feasibility of using polymer films based on polyvinyl alcohol and chitosan to clean oil-contaminated soils.

Keywords: polyvinyl alcohol, chitosan, glycerol, polymer films, oil-contaminated soils, oil products, phytoremediation, remediation.

Introduction

Soil belongs to the most important natural resources, the state of which mostly provides the ecological equilibrium of the planet. The main characteristic of the soil is fertility, which is formed by the microorganisms' activity. Economic activity contaminates oil, that leads to a decreasing in economic and potential fertility [1]. Soil pollution by oil and oil products is currently an actual problem. Oil production from the bowels, processing and transportation are not only technically difficult, but also dangerous processes, since it is impossible to preserve the natural environmental conditions during field development. Inevitably, each stage of oil production is accompanied by an oil leak, which can cause irreparable phenomena. Chronic oil spills pose a serious threat to the environment and human health [2].

Restoration of the environment with the help of plants is of wide interest due to the opportunities that this technology opens up in the cleaning of contaminated areas. Over the past decade, phytoremediation has become very popular, as this technology is much cheaper than methods based on the application of technology, and is also used directly in the area of pollution [3].

The purpose of this work is to study the possibility of using composite materials based on polyvinyl alcohol and chitosan for purification of oil-contaminated soil in the process of phytoremediation.

Experimental

In our work we used polyvinyl alcohol (PVA) with molecular weights 72,000 and 145,000 kDa, chitosan (CT) produced by «Fluka Chemie GmbH» (Switzerland), acetic acid and glycerin from «SCAT» (Kazakhstan) without additional purification. The synthesis of polyvinyl alcohol and chitosan copolymers is described in work [4]. In this article, initial polymer solutions are prepared in advance to obtain films based on a mixture of polyvinyl alcohol and chitosan. To do this, in distilled water, the PVA suspension is dissolved at a temperature of 70–75 °C, the chitosan suspension is dissolved in acetic acid at room temperature. After complete dissolution of chitosan, a cooled PVA solution was introduced into the solution with constant stirring to homogenize the solution. After a certain time, continuing mixing, glycerin was added as a plasticizer. Before molding, the resulting solution was kept at room temperature for 10 to 12 hours for de-airing and stabilization. The films were formed in Petri dishes by evaporation of the solvent at room temperature for 1–2 days under the drying cabinet. The mass of the films was 7–8 g. The films were obtained at different ratios of polyvinyl alcohol/chitosan — 20:80; 50:50 and 20:80 wt.%.

Organic solvents (acetone, ethyl alcohol, benzene, a mixture of alcohol: benzene in a 1:1 ratio) and distilled water were used to study the solubility of films.

The IR spectra of the obtained films were recorded on an IR spectrometer with a Fourier Converter («LOMO», Russia).

The mechanical properties of the films (tensile strength) were studied on a discontinuous machine «TEXTURE ANALYSER» (UK) with a break rate of 10 mm/min, with a force of 20,000 kg.

To study the phytoremediation process, the experiment was carried out under laboratory conditions in 500 ml polyethylene cups filled with oil-contaminated soil (the oil content was 15g/500g, 30g/500g, 45g/500g % of the total mass). In the experiment we used the oil from the Karazhanbas field.

To determine the oil content in the soil, we used the extraction method on the Soxlet apparatus. Extraction of petroleum hydrocarbons from contaminated soil samples were performed after 60 days of their interaction with each other. Benzene was used as a solvent. The flask with the solvent is heated to the boiling point of the solvent, the vapors of which enter continuously into the cooler, into the ground mixture, dissolve the hydrocarbon part and secrete from the mixture. After filling the extraction flask with a siphon tube, the solvent flows in enters the flask. Before the solvent color is lost, the organic part is extracted. The sample from the extractor is dried in a drying cabinet to a constant mass at a temperature of 60–70 °C. The mixture of the organic part of the solvent and the oil residue is separated by simple distillation here.

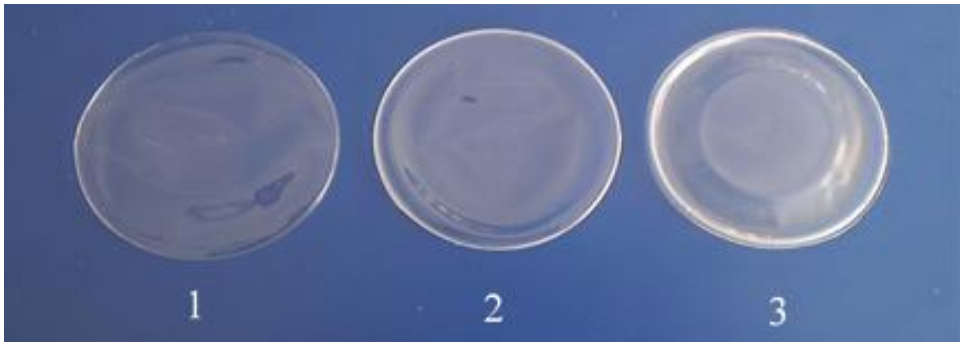
Results and Discussion

Hydrophilic polymers, which include polyvinyl alcohol (PVA), are widely used in medicine, biochemistry, biotechnology. The possibility of their modification, including by mixing with other polymers, contributes to the expansion of areas and efficiency of application of hydrophilic polymers. The most promising are mixtures of PVA with the natural polysaccharide chitosan, which can be obtained in the form of films from a common solvent. It is established that this leads to an increase in the stability of the films, improves their physical and mechanical parameters, etc.

In the proposed work, films based on polyvinyl alcohol and chitosan are obtained by mechanical mixing of specified ratios of components, glycerin is used as a plasticizer. The ratio of the initial polymers was varied in the preparation of films. Thus, films with the content of PVA/CT = 20:80, 50:50 and 20:80 wt.% were obtained, the uniformity and transparency of which indicates the compatibility of the original components. Moreover, an increase in the chitosan content in the initial polymer mixture affects the transparency: less transparent films are formed. Pictures of the films are shown in Figure 1.

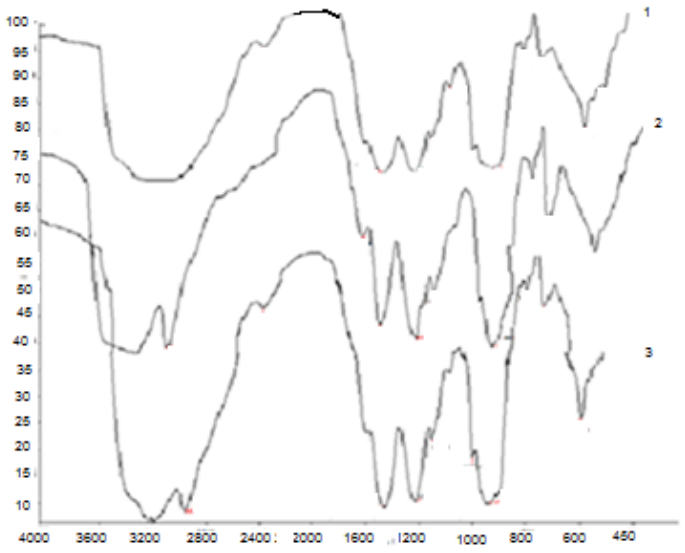
The compatibility of the initial components is indicated by the IR spectra of films based on PVA/CT, presented in Figure 2 and in which you can see the presence of the composition of the two components of the film. It is known that absorption bands of NH_2-groups are present on the IR spectra of chitosan, as well as absorption bands characteristic of the polysaccharide structure at 1,050 cm^{-1} and 851 cm^{-1} [5]. Intense fluctuations at 1,645 cm^{-1} and at 1,565 cm^{-1}, characteristic of the amide groups, indicates that the initial chitosan has a high degree of deacetylation. In the spectra of PVA/CT mixtures, there are changes in the position and intensity of the absorption bands: the presence of a weak absorption band of the carbonyl C=O group at 1,735 cm^{-1} in the PVA spectrum indicates that part of the OH groups of the polymer is acetylated during dissolution in acetic acid solution. Judging by the low intensity of this band and the high intensity of the OH-group band (947 cm^{-1}), the degree of acetylation is very insignificant. An increase in the chitosan content in the mixture

causes a gradual shift of the absorption band C=O of the PVA groups at 1,735 cm^{-1} (this band is absent in the chitosan film) to the lower frequencies. This may suggest a possible interaction between the C=O groups of partially acetylated PVA and the active groups of chitosan. From the analysis of IR spectra, it follows that the interaction of polyvinyl alcohol and chitosan leads to molecular interaction between the hydroxyl groups of alcohol macromolecules and the amide groups of chitosan, and hydrogen bonds are formed according to the scheme below.



1 — 80:20; *2* — 50:50; *3* — 20:80 wt.%

Figure 1. Films based on PVA/CT at different ratios of the initial components



PVA/CT: *1* — 20:80; *2* — 50:50; *3* — 80:20 wt.%; Mw[PVA] = 145,000

Figure 2. IR spectra of films based on PVA/CT

The solubility of these films in distilled water and in organic solvents was studied. The dissolution process was carried out at room temperature for 24 hours. This study showed that films based on PVA/CT in organic solvents such as acetone, ethyl alcohol, benzene, alcohol-benzene mixture, do not dissolve in distilled water organic solvents. This is probably due to the impossibility of breaking the bonds between the polymers in the macromolecule. Since organic solvents are unable to break the bonds of polymer macromolecules in the model. While these polymers are dissolved in distilled water during the day due to the processes of solvation and hydration. The effect of the molar mass of PVA on the solubility of films based on PVA/CT was established. Thus, films with a molecular weight MW(PVA) = 72,000 are dissolved within 18 hours, and the solubility of films based on PVA with a molecular weight of 145,000 is 24 hours (Table 1).

Table 1

Results of mechanical stretching of the films of PVA/CT

Parameter	Film ratio = PVA/CT, wt.%					
	20:80		50:50		80:20	
M_w (PVA)	72,000	145,000	72,000	145,000	72,000	145,000
Pressure, MPa	37.44	61.78	22.21	33.76	57.60	29.47
Stretching, %	4.63	18.95	47.32	40.37	116.75	198.12
Young's modulus, kPa	1.9±5	1.4±5	2.3±5	2.0±5	2.9±5	2.9±5

The mechanical properties of the obtained films were investigated. All obtained films have good mechanical strength. An increase in chitosan content leads to a decrease in elongation and an increase in the modulus of elastic, and as a result, to an increase in brittleness. The highest rates of mechanical parameters were shown by films containing 80 % synthetic polymer — PVA. The latter is apparently due to the formation of a grid of intermolecular hydrogen bonds between chitosan and PVA.

The issues of remediation of oil-contaminated soils are relevant in connection with the rapid development of oil and gas industry and infrastructure [6]. Increasing oil production continues to increase the risk of soil contamination with oil. Every year, oil-contaminated areas expand, and land used for agriculture and forestry remains unusable. Due to the high adsorption, oil can be observed in the soil for a long time, shifting the soil balance from the equilibrium level. Plants are the key contributor to the high fertility of the soil. However, unfavorable growing conditions on oil-contaminated soils of any plants, excludes the possibility of cleaning such soils by phytoremediation. Natural polymers are not xenobiotics and their use is the most environmentally and economically rational for exosystems.Their low cost and simple preparation technology together with high performance properties make it possible to effectively clean the soil from oil products.

Chitosan is a natural biopolymer, an aminopolysaccharide, which has no pronounced substrate specificity, which means approximately the same ability to bind both hydrophilic and hydrophobic compounds. Chitosan has ion-exchange, chelation-forming and complex-forming properties [7]. Chitosan is similar in composition to nonspecific organic compounds of soils, this eliminates the possibility of a negative impact on the processes of decomposition, synthesis of organic compounds, humus formation.

We studied for the first time the possibility of using synthetic films based on PVA/CT for soil detoxification with simultaneous planting of technical crops.

Modeling of biodegradation of oil products was carried out at temperatures of about 25–30 °C, simulating the temperature regimes of the summer months in Atyrau, Mangistau and West Kazakhstan regions. The soil selected within Almaty was subjected to researches. Soil sampling was carried out in accordance with State Standard 7.4.3.01–831. The soil was freed from stones and roots of plants, dried in the absence of sunlight to an air-dry state, sifted and placed in open containers. Pollution modeling was carried out by means of introduction of oil of the Karazhanbas field into the soil and according to gradation of soils on degree of pollution with oil products (OP) developed by V.S. Khomich [8], introducing oil before achievement of mass concentration of oil products in the soil of 15g/500g, 30g/500g, 45g/500g that corresponded to the category «very strongly polluted soils». The effectiveness of remediation of soils contaminated with oil products was estimated by measuring the content of oil products and phytotesting of studied soil samples.

Phytotesting is based on the sensitivity of plants to exogenous chemical effects, which affects the growth and morphological functions of test cultures. Dry seeds of the selected test cultures were placed at intervals of 1 cm in polyethylene cups (with mixed and moistened soil samples). As a control, samples of the studied soil without the introduction of oil products were used. As indicators of the phytotest, test functions of the height of seedlings were adopted.

The dynamics of changes in the concentration of oil products was evaluated by the method of their extraction from contaminated soils. The analysis of quantitative composition of hydrocarbons of the Karazhanbas oil was carried out on the soxlet apparatus. Analysis of changes in oil content in the studied soil samples showed that during the first two weeks, the level of oil dropped significantly compared to the original values as in the control sample (to 25.6 %) and in samples containing OP, which is obviously related to the first phase of degradation of oil products evaporation of light fractions and photooxidation. At the same time, in soil samples containing films based on PVA/CT, the loss of oil products on day 14 was 36.0 %.

After 14 days of the experiment, the achieved concentration of petroleum products in the control soil sample (which does not contain petroleum products) remained almost unchanged until the end of the

experiments. In soil samples with films, there is a decrease in the concentration of petroleum products. This, apparently, indicates the end of the process of degradation of petroleum products in these conditions.

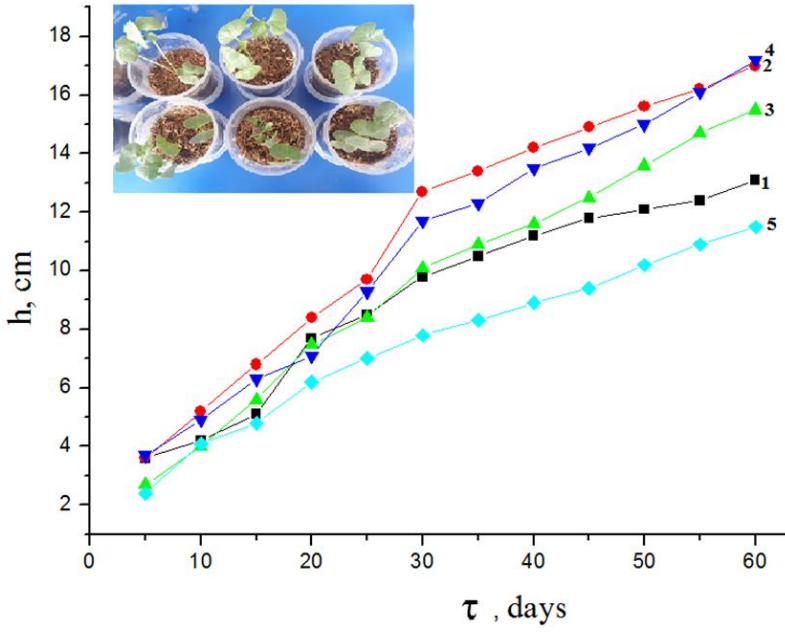
In soil samples with the addition of films based on PVA/CT, the dynamics of reducing the concentration of oil products up to 21 days was approximately 40 %. It can be noted that the degradation of oil hydrocarbons was accelerated by films based on PVA/CT, increasing the efficiency of soil purification from oil products in comparison with the self-cleaning ability of the soil, the level of which was 25.6 %. In addition, the work assessed the degree of soil purification from oil products. The data are presented in Table 2.

Table 2

The purification degree of soils contaminated with oil

Concentration of oil products in soil samples, g/500g	Polymer concentration, g	Purification degree, %
15	0.1	86.9
30	0.1	82.3
45	0.1	78.7

In the process of transformation of oil products, intermediates are formed, which no longer fall under the definition of «oil products» and are not taken into account in the gross definition of OP. At the same time, these compounds can have a significantly greater toxic potential and have a positive ecotoxic effect. Carrying out ecotoxicological phytotesting in addition to the instrumental method of assessing the content of OP in the process of remediation allows to quickly and fully assess the environmental quality of the soil environment [9]. Assessment of soil samples by indicators of test functions of test crops Figure 3 established the effectiveness of using films based on PVA/CT as recultivants to reduce the level of oil pollution in the studied soil samples.



1 — soil; *2* — soil with films; *3* — 15g/500g; *4* — 30g/500g; *5* — 45g/500g

Figure 3. Length of test plant seedlings in soil samples

According to the results of the study, the indicator «seedling length» for control soil samples without films after 60 days was 10 cm in the case of reclamation of soil samples using films without refining waste, the length of the seedling on day 60 of the study reached 17 cm after 60 days, the least phytotoxic effect (along the length of the seedling) was observed for soil samples using films based on PVA/CT with a maximum content of OP.

The maximum length of the seedling is typical for soil samples uncontaminated by oil waste containing films based on PVA/CT. Slightly different indicators for soil samples contaminated with oil products.

Probably, due to the presence of free amino groups in the macromolecule of the polymer, chitosan effectively clears the soil, which is explained by the process of complexation of oil products by amino groups, with the formation of polynuclear compounds. Also because of the abundance of hydrogen bonds between chitosan molecules — not soluble in water, but swells in organic acids, in the swollen state is able to firmly hold various intoxicants (pollutants).

The studies suggest that films based on polyvinyl alcohol/chitosan will be widely used in solving the problems of cleaning soils contaminated with oil products. When PVA/CT films are introduced into the soil as a remediant, they do not need to be collected and disposed of, as they are capable of undergoing enzymatic degradation, increasing the level of natural soil carbon.

Conclusions

We obtained films based on polyvinyl alcohol and chitosan. By IR spectroscopy it is shown that polyvinyl alcohol and chitosan form a mixture stabilized by a hydrogen bond. It is established that with increasing content of polysaccharide in the composition of the film, their strength properties decrease. The results of the assessment of degradation of oil products in the process of reclamation and phytotesting indicators obtained in the dynamics of the experiment allow us to conclude about the effectiveness and feasibility of using polymer films based on polyvinyl alcohol and chitosan as recultivants of oil-contaminated soils.

Acknowledgment

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Поливинил спирті негізіндегі композициялық материалдарды топырақ фиторемедиациясында қолдану

Мұнайды өндіру, тасымалдау, ауыстырып тиеу және сақтау топырақтың мұнай өнімдерімен авариялық ластануына әкеп соғады. Осы мәселені шешу барысында зерттеушілер мұнаймен ластанған топырақты қалпына келтіру үшін полимерлі үлдірлерді қолдану мүмкіндігін ұсынған. Мақалада топырақты мұнай

өнімдерінен тазартуда қолдану мақсатында поливинил спирті мен хитозан негізінде үлдірлер алынған, олардың негізгі физика-механикалық қасиеттері зерттелген. Зерттеуде ИК-спектрометриялық талдау нәтижелері бастапқы компоненттердің жақсы үйлесімділігін көрсетті. Алынған үлдірлер жоғары механикалық беріктік көрсеткіштерге ие екені анықталған. Сонымен қатар, механикалық параметрлердің барынша жоғары көрсеткіштерінің құрамында 80 % синтетикалық полимер-ПВС бар екенін үлдірлер көрсетті. Бұл жағдай хитозан мен ПВС арасындағы молекулааралық сутегі байланыстары торының түзілуімен байланысты болуы мүмкін, бұл олардың қолданылу сипаттамаларына жағымды әсер етеді. Ластануды модельдеу Қаражанбас кен орнының мұнайын топыраққа енгізу арқылы жүргізілді. Поливинил спирті мен хитозан негізіндегі алынған үлдірлер 0,2 г/кг топырақ концентрациясына енгізілген рекультивант ретінде қолданылды. Мұнаймен ластанған топырақты ремедиациялау тиімділігі мұнай өнімдерінің құрамын өлшеу және зерттелетін топырақ үлгілерін фитотестілеуді жүргізу барысында бағаланды. Қайта құнарландыру процесінде мұнай өнімдерінің ыдырауын бағалау нәтижелері және эксперимент динамикасында алынған фитотестілеу көрсеткіштері поливинил спирті мен хитозан негізіндегі полимерлі пленкаларды мұнаймен ластанған топырақты тазалау үшін пайдаланудың тиімділігі мен орындылығы туралы қорытынды жасауға мүмкіндік береді.

Кілт сөздер: поливинил спирті, хитозан, глицерин, полимерлі үлдірлер, мұнаймен ластанған топырақ, мұнай өнімдері, фиторемедиация, ремедиация.

Д.Т. Абдулетип, П.И. Уркимбаева, Г.А. Мун, З.А. Кенесова, Б. Хавилхайрат

Применение композиционных материалов на основе поливинилового спирта в фиторемедиации почв

Добыча, транспортировка, перевалка и хранение нефти приводят к аварийным загрязнениям почв нефтепродуктами. При решении данной проблемы исследователями рассматривалась возможность применения полимерных пленок для рекультивации нефтезагрязненных почв. В настоящей работе с целью очистки почвы от нефтепродуктов получены пленки на основе поливинилового спирта и хитозана, исследованы их основные физико-механические свойства. Результаты ИК-спектрометрического анализа показали хорошую совместимость исходных компонентов. Обнаружено, что полученные пленки обладают достаточно высокими показателями механической прочности. Причем максимально высокие показатели механических параметров показали пленки, содержащие 80 % синтетического полимера — ПВС. Последнее, видимо, связано с образованием сетки межмолекулярных водородных связей между хитозаном и ПВС, что благоприятно влияет на их эксплуатационные характеристики. Моделирование загрязнения проводилось посредством внесения в почву нефти Каражанбасского месторождения. Полученные пленки на основе поливинилового спирта и хитозана использовали в качестве рекультивантов, которые вносились в концентрациях 0,2 г/кг почвы. Эффективность ремедиации загрязненных нефтью почв оценивалась в ходе измерения содержания нефтепродуктов и проведения фитотестирования исследуемых образцов почв. Результаты оценки деградации нефтепродуктов в процессе рекультивации и показатели фитотестирования, полученные в динамике эксперимента, позволяют сделать вывод об эффективности и целесообразности использования полимерных пленок на основе поливинилового спирта и хитозана для очистки нефтезагрязненных почв.

Ключевые слова: поливиниловый спирт, хитозан, глицерин, полимерные пленки, нефтезагрязненные почвы, нефтепродукты, фиторемедиация, ремедиация.

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