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# Regeneration of Sorbents Mixture After the Purification of Recycled Water in Production of Soft Drinks

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**Abstract**—The regeneration of used mixed sorbents (activated carbon with kieselgur) for their repeated usage in the purification of recycled water from organic impurities during the production of soft drinks has been presented in this study. The sequence of reagent interaction and the staging of acid–base processing of used sorbents allowed recovering 100% of their sorption capacity. The schematic of a three-stage adsorption device for purification of industrial water from organic impurities has been proposed and the possibility of its operation in closed circulating cycles of industrial production has been shown.

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*Keywords:* regeneration, sorbent, industrial water, organic impurities, plant, purification, recovery.

## INTRODUCTION

Modern technologies of the food industry should be able to solve complex issues of power-, resource- and ecology conservation [1]. According to this, the production of the soft drinks should provide for the following interconnected technological processes: additional purification of processed water with an increase of it part in industrial cycles [2]; additional use of filtering substances (activated carbon (AC), kieselgur (K), other artificial or natural sorbents) [3]; development of module technologies when using multistage absorption units with subsequent (staged) introduction of the sorbent [4].

Recycling of spent sorbents and processed water could diminish the costs and, as a result, decrease the ecological load on the environment. Aqueous sugar syrup which remains after the production of soft drinks can be recycled too. Despite the great number of modern methods of water purification from organic impurities such as nano-, ultra-, microfiltration, coagulation, reagent treatment and photocatalysis [5], adsorption by AC remains one of the most effective methods. However, a large amount of AC and other sorbents, which are used in the food industry, are not recycled because of the complexity of their regeneration and great material costs. Therefore, the purpose of this research was to study how to regenerate the mixture of spent sorbents (AC and K) and use them for purification of technical recycled water in the production of soft drinks.

## EXPERIMENTAL

In carrying out experiments we used the spent sorbents mixture (Panda enterprise, Vinnitsa), which consisted of AC (Decolar A) and kiselgur (Bekogur 3500) in a weight ratio 4 : 6, and which was placed on the supportive cardboard (INDURA) of a filer–press.

Organic impurities were removed from the sorbents mixture in the process of their regeneration using a laboratory unit, which consisted of a magnetic mixer VELP AREC (VELP Scientifica, Italy), a flat bottom flask and Liebich Refregirator. 100 g of spent sorbent mixture was mixed with 400 $cm^3$ of distilled water, followed by stirring on a magnetic mixer (250 rpm) at 50–60°C for 45–60 min. Then the reaction mass was cooled down to an ambient temperature, filtered, and then the above-described procedure was repeated one more time. In this case, the reaction mass was rinsed with 600 $cm^3$ of water followed by purification of water filtrate by earlier regenerated sorbents mixture. After the first stage of regeneration, the sorbents mixture was dried to a constant mass at 104°C, then 100 $cm^3$ of a 1% solution of NaOH was added, and the mixture was boiled for 45–60 min. Then the reaction mass was cooled, filtered and subjected to the subsequent treatment with

100 $cm^3$ of a 4% solution of HCl under the same conditions. Water filtrates of the second stage of regeneration (200 $cm^3$) were combined and neutralized to pH 7.

The purification of water filtrate (F-I) after the first stage of the regeneration of the sorbents mixture was carried out on the same laboratory unit. 10 g of sorbent was added to 200 $cm^3$ of water filtrate, stirred on the magnetic mixer at 200 rpm for 6 min. The sorbent was separated by filtration and resultant filtrate was treated in the same way two more times.

Determination of the sorption capacity of the sorbents mixture in terms of iodine was conducted according to the established technique [6]. Measurements have been performed in duplicate, arithmetic mean was calculated, the absolute error of the mean should not exceed 3%.

The residual amount of sugar (glucose) in water filtrate was determined on a refractometer 1RF 454 62 M by the indicator of refraction of the studied solutions [7].

The X-ray phase analysis of sorbents mixture (AC and K) was conducted on a diffractometer (Drone-2) in a monochromatized Co-K$\alpha$-radiation ($\lambda = 1.79 \times 10^{-1}$ nm). Components were identified by way of comparison of interfacial distances ($d$, nm) and relative intensities ($I_{rel}$–$I/I_0$) of the experimental curve with the data of electronic file PCPDFWIN [8].

## RESULTS AND DISCUSSION

The efficiency of regeneration of spent sorbents mixture (AC and K) was determined by the change of their overall sorption capacity. The obtained data shows that the sorption capacity of regenerated sorbents mixture treated either with a solution of NaOH or HCl increased by 29–42% and 23.8–29.0% compared to a spent sorbent, respectively. The most effective result was achieved when using a 1.25% alkaline solution and by joint treatment of the sorbents mixture with solutions of NaOH and HCl. In this case, the sorption capacity of regenerated sorbents increased by 42% and reached the initial value before technological application for the purification of sugar syrup (Fig. 1).

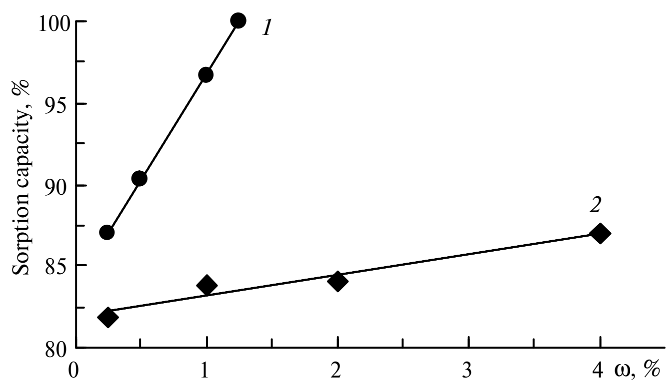


**Fig. 1.** Dependence of the sorption capacity of the regenerated mixture of sorbents (AC and K) from the concentration of solutions: *1*—NaOH; *2*—HCl.

Presumably, such essential differences of regeneration efficiency of the sorbents mixture can be explained by the presence of absorbed substances of different chemical nature: coal ash, melanoidins, and products of their chemical transformation [9]. It is generally accepted that the regeneration efficiency of AC after the treatment by a 1–4% solution of chloride acid depends on the removal of coal ash, however, our data (see Fig. 1) show that the loss of sorption activity of the sorbents mixture does not depend on the presence of coal ash, but is determined by absorbed melanoidins and their metabolites. A confirmation of this fact was obtained by the authors [10], who demonstrated that melanoidins are resistant to hydrolysis by concentrated mineral acids. Meanwhile, it was established that the treatment of the spent sorbents mixture by a solution of NaOH results in a complete restoration of their sorption capacity (see Fig. 1). Adsorbed organic impurities and dyes can chemically interact with NaOH forming water-soluble salts that could be actively detached from a solid surface [11] increasing, in this case, a sorption capacity of a sorbent.

To investigate changes in the solid phase composition of a sorbents mixture we used X-ray diffraction spectroscopy, which was also effectively applied in the investigation of other similar adsorption systems [12]. Thus, the data of the X-ray phase analysis (Fig. 2) confirmed the amorphous state of activated carbon and the presence in the left-hand part of diffractograms of kiselgur of the main peak of crystabolite $SiO_2$ which is located at 26°C and well characterize the initial (diffractogram *1*) and regenerated mixture of sorbents (diffractogram *3*). However, on the diffractogram *2* of spent sorbents mixture, this peak is shifted to a lower value of diffraction angles, from 26 to 23°. Thus, the position of the main peak of crystabolite $SiO_2$ in the diffractogram may be considered a reliable marker, which could indicate the efficiency of regeneration of the spent sorbents mixture.

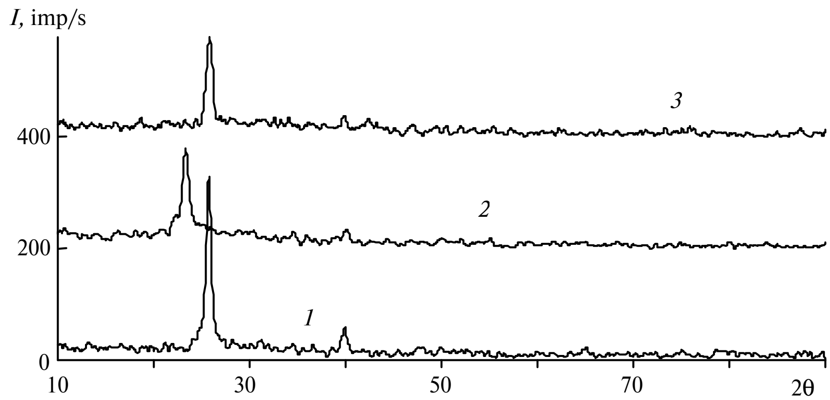


**Fig. 2.** Diffractograms of the mixture of sorbents (AC and K): *1*—initial sample; *2*—a sample washed by water; *3*—regenerated sample. Diffractograms *2, 3* shifted along the *Y*-axis by 200 imp/s.

Next, we determined the possibility of using a regenerated sorbents mixture for the purification of contaminated by organic impurities water, which was collected after the first stage of the regeneration of these sorbents. The research was carried out on a laboratory unit using the principle of a three-stage adsorption unit with subsequent use of the regenerated sorbents. The data obtained in this case with respect to the main content of sugar are given in the table and the proposed technological flow chart of purification of the processed water is shown in Fig. 3. We established the possibility of the three-fold regeneration of the mixture of sorbents, which corresponds to 15 cycles of purification of initial sugar syrup (the Working instruction ROB 066.01-06 PF Panda). The content of sugar, the main contaminant of process water, decreased in 2.9 times per one cycle, which points to the high sorption efficiency of the regenerated sorbents. The purification unit includes three mixers–collectors, pumps and filters for water filtration. Purified water (F-IV) could be used for the recycled water supply of the technological cycle or could be purified additionally. Such a process ensures a decrease in total water consumption in the production of soft drinks.

Characterization of the stepwise purification of the primary filtrate F-I

| Investigated solution | Solution which was subject to adsorption sampled after | Refraction indicator | Sugar content |
|---|---|---|---|
| F-I | Washing with water of the spent mixture (AC and K) | 1.3350 | 0.060 |
| F-II | Mixer–collector *1* | 1.3347 | 0.050 |
| F-III | Mixer–collector *1′* | 1.3342 | 0.038 |
| F-IV | Mixer–collector *1″* | 1.3337 | 0.021 |

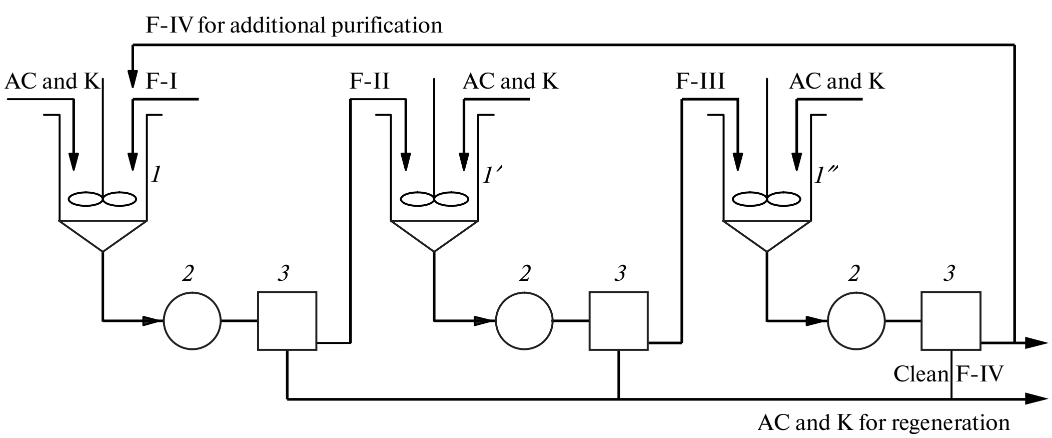


**Fig. 3.** Schematic of the three-stage adsorption unit with the subsequent introduction of filtrate: *1, 1′, 1″*—mixers-collectors; *2*—pump; *3*—filter [4].

The conducted research upon regeneration of the mixture of sorbents (AC and K) and their repeated use for purification of process water obtained after the production of soft drinks provides a solution to the complex of ecological and resource-saving issues within the framework of one industrial production.

## CONCLUSIONS

Based on the obtained data we determined the possibility of 100% regeneration of the sorption capacity of the spend sorbents mixture (AC and K) by its subsequent acid-base treatment. We also established the possibility of using the three-stage adsorption unit in a combination with regenerated sorbents for purification of process water which was obtained after the production of soft drinks. This approach provides the possibility of nearly up to three folds to decrease organic contaminants in process water.

## REFERENCES

1. Seminozhenko, V.P., Stalinskaya, D.V., and Kasimov, A.M., *Promyshlenye otkhody: problemy i pyti resheniya* (Industrial Wastes: Problems and Ways of Solving), Kharkiv: Industriya, 2011.
2. Sorokin, A.I., *Oborotnoe vodosnabzhenie sakharnykh zavodov* (Recycling Water Supply of Sugar-Fabrication Plant), Moscow: Agropromizdat, 1989.
3. Oliinyk, S.I., Prybilskii, V.L., Kuts, A.M., Kovalchuk, V.P., and Kovalenko, O.O., Aktyvne vugillya u vodogotuvanni dlya vygtuvannya napoiv, *Kharchova Nauka i Tekhnologiya*, 2014, vol. 28, no. 3, pp. 36–42.
4. Proskuryakov, V.A. and Shmidt, L.I., *Ochistka stochnykh vod v khiecheskoi promyshlennosti* (Purification of Wastewaters in Chemical Industry), Leningrad: Khimiya, 1977.
5. Kochkodan, V.M., Rolya, E.A., and Goncharuk, V.V., Photocatalytic membrane reactors for water purification from organic pollutants, *J. Water Chem. and Technology*, 2009, vol. 31, no. 4, pp. 227–237.
6. GOST 6217-74. Ugol aktivnyi drevesnyi. Technicheskie usloviya (s izmeneniyami no. 1, 2, 3, 4), introduced on January 1, 1976 (Wood Charcoal, Technical conditions (with additions no. 1, 2, 3, 4).
7. GOST R 50546-93. Glukose Sirup. Opredeleniye spderzhaniya sukhogo veshchestva s ispolzovaniem porkazatelya prelomlenya (Determination of the content of dry substance with the use of reflection indicator. Refractometric method. Introduced on January 1. 1994).
8. JCPDS-PDF database, International centre for Diffraction Data 1999, PCPDFWIN, v. 2.02.
9. Tarasova, I.A., Sovershenstvovanie adsorrbtsionnoi ochistki sakharosoderzhashchikh rastvorov, *Cand. Sc. (Techn.), Dissertation*, Moscow State. Univers. of Pishchevoi Promyshlenosti, Moscow, 1969.
10. Kuzin, A.M., *Oshchaya biotekhnologya* (General Biotechnology), Moscow: Vyssh. Shkola, 1969.
11. Shtagneev, V.O., and Molodniskaya, E.N., Ochistka gustykh produktov sakharnogo proizvodstva, *Sahar*, 2013, no. 11, pp. 4–49.
12. Zamatyrina, V.A., Metod ochistki stochnykh vod ot tyazhelykh metallov i neftepproduktov s ispolzovaniem modifitsirovanogo organobentonita (Method of Purification of Wastewaters of Heavy Metals and Petroleum Products with the Use of Modified Organobentonite), *Cand. Sc. (Techn.), Dissertation*, FGBOU VPO, Saratov. State Tech. Univer. im. Gagarin, 2015.

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