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Optimal evaporation regime of alkaline solution obtained from cotton stalk ash and chemical analysis of precipitation process

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ABSTRACT

This article describes a comprehensive analysis of the optimal evaporation method for an alkaline solution that is prepared using the aqueous extract of cotton stalk ash, along with the investigation of the chemical composition of the precipitation that appears during evaporation of the solution. Cotton stalk ash is regarded as a secondary product from burning biomass in household appliances, specifically in tandir ovens used to bake bread and somsa, and is currently being discarded into the environment. In the presented research, the procedure for preparation of the alkaline solution was conducted under optimal conditions, when the mixture of ash and water with the weight ratio of 1:5 was boiled. Then, filtration and evaporation of the obtained suspension were performed. From the results obtained, the optimal evaporation regime was identified as 75%, as during this regime no precipitation occurs. Nevertheless, at an evaporation level of 80%, precipitation was observed in the solution due to the disturbance of the solubility equilibrium. The content of the precipitated components in relation to cations was estimated using flame photometry, and the phase composition was evaluated with FTIR spectroscopy. Thus, the precipitate was found to contain mainly carbonate-like compounds. It was proved that potassium ions prevailed in the solution, and their molar concentration is much greater than that of other cations. Moreover, when the concentration is high, a certain share of potassium ions is transferred from the solution into the precipitate phase. As calculated, the total alkalinity of the prepared solution equals 3.41 N and constitutes about half of the alkalinity of the 6 N NaOH solution employed in the vegetable oils purification process.

Keywords: cotton stalk ash, extraction, evaporation, potassium ions, carbonates, precipitate, flame photometry, FTIR, co-precipitation, alkaline solution.

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Introduction

Rational and effective use of waste from plant biomass has become one of the priority trends in scientific research. The resulting ash from burning plant biomass consists of a complex mineral composition, where potassium, calcium, and silicon compounds play an important role as predominant elements. There is a considerable difference in the proportion of these elements, depending on the

type of raw materials used and the conditions of the burning process [[1], [2], [3], [4], [5]].

Based on existing scientific publications, it is possible to state that potassium-containing compounds in biomass ash have good solubility in water and transfer to the solution phase during extraction and play the role of active agents. Calcium compounds, in turn, are found mainly in the insoluble carbonate form and do not dissolve in aqueous solutions [[6], [7]].

Extraction of alkaline components from the ash involves a series of complex physicochemical processes, including ion exchange, solubility of substances, and phase equilibrium processes. Due to the high solubility of potassium and sodium salts in water, they selectively dissolve in an aqueous phase, thus forming a high concentration of alkali solution [[8], [9]].

The physicochemical properties of the obtained solution are determined by several main technological parameters: temperature, duration of the extraction process, and the ratio between the liquid and solid phases. The aforementioned technological parameters have a significant impact on the formation and stabilization of the composition of the solution [8].

Technologically, it is important to control the evaporation process and obtain an aqueous solution with high concentration. It should be noted that the higher the concentration of the solution, the higher its degree of alkalinity; but beyond a critical point, the solubility equilibrium is disturbed, leading to precipitation of poorly soluble products of ion reactions [[10], [11].

In conditions of high concentration, the interaction between the ions increases, causing co-precipitation reactions. It contributes to the creation of poorly soluble complexes and alloys, reducing the compositional stability of the solution and causing the loss of a part of the required substances. Thus, setting the required level of evaporation and its effective regulation become essential aspects when striving to improve the performance characteristics of the technological complex [[11], [12]].

The alkaline solution produced based on biomass ash is expected to be a prospective compound applicable in many industrial procedures. For example, in processing vegetable oil, it has a significant function of neutralizing free fatty acids [[13], [14], [15]].

The success rate of the procedure in question largely depends on the concentration and chemical composition of the alkaline solution, and both parameters play a critical role in the effectiveness of neutralization and the quality of the end-product [16]. In addition, the feasibility of utilizing such solutions as an alternative agent instead of traditional sodium hydroxide is justified in the literature as well [3].

From research findings, it can be concluded that biomass-based ash materials, especially plantain (heat-treated edible banana) peel ash, and in the mixture with traditional adsorbents, can efficiently reduce the concentration of free fatty acids (FFA) in vegetable oil. Under optimized conditions, the maximum efficiency of removing FFA through using these materials may range from 70% to 97% [3].

The high efficiency of the method can be attributed to the strong reaction of active substances in the ash with FFA and excellent adsorption properties of the material.

The process of obtaining potassium carbonate from cocoa pod ash has also been studied, where the major impurity element has been revealed to be calcium ions. It was additionally noted that the efficient removal of such impurities from the solution is crucial [1].

Thus, the above data prove the feasibility of the use of alkaline compounds isolated from the biomass ash as an alternative or complementary substance for conventional alkaline reagents in the vegetable oil refining process, especially in neutralizing them. At the same time, this approach is advantageous not only due to its high efficiency but also because it lowers production expenses.

The technology of processing vegetable oils involves several consecutive steps, each one aimed at removing specific impurities from vegetable oils and concluding according to further processing needs. The allowed content of accompanying components of vegetable oils is defined by special standards and requirements, depending on the nature, quality, and application of vegetable oils.

Free fatty acid content in vegetable oils negatively influences the sensory qualities and increases oxidation rate. In addition, there have been proven dependencies between accompanying substance content and composition and ripeness of vegetable seeds used for oil production as well as processing conditions [[17], [18], [19]].

In processing vegetable oils with caustic soda, some part of carotenoids is absorbed by particles within the soap stock phase, which appears due to intensive interaction of oil with highly concentrated alkaline solution. Nevertheless, most of them are eliminated by solid adsorbents, which have an

elaborate structure [[20], [21], [22], [23], [24], [25], [26]].

Thus, the data presented above show that alkaline solutions derived from biomass ash are distinguished by their pronounced alkalinity and have significant prospects for the use of these solutions in industrial processes, including refining vegetable oils [[1], [3]]. Nevertheless, while the process of extraction of ash solutions is described thoroughly in previous scientific research, the further technological step-the process of evaporation and its impact on the composition of the solution and precipitation-is insufficiently studied. Specifically, the issues of ions' redistribution and their transformation in the solid state during the process of high concentration of the solution demand further scientific research.

As recent scientific research has revealed, the alkaline solutions derived from biomass ash may serve as an alternative source of sodium hydroxide in the industry. Such a method provides effective recycling of production wastes, allows decreasing the amount of imported chemicals, and helps use resources rationally [[3], [27], [28], [29]].

Consequently, in the current study, the most effective regimes for the evaporation of the alkaline solution prepared from cotton stalk ash were found, and a detailed analysis of ions' distribution in the solution and precipitate and their chemical properties were carried out using the flame photometry and infrared spectroscopy approaches. However, since the composition and concentrations of the alkaline solutions have a huge impact on the total effectiveness of the purification process, there should be some research conducted in this field. Indeed, it can be noted that such factors as the influence of evaporation on the solution's composition and the content of the active substance have not been considered thoroughly enough yet.

Materials and Methods

As the subject for investigation, the solution derived through the aqueous extraction of cotton stalk ash, along with the precipitates derived through the evaporation of said solution, were chosen. As the first step of the experiment, cotton stalk ash was combined with water in different mass ratios and heated up to dissolve the soluble compounds in the liquid phase. According to the results of the experiments conducted, the highest concentration of compounds in the solution was achieved using an ash-to-water mass ratio of 1:5.

This solution was filtered to separate the solid mass from the solution, and then the filtrate was kept separately and was evaporated to different degrees. In terms of filtrate evaporation, the conclusion is that evaporation up to 75% of the solution can be considered an optimal stage. In the case of such evaporation, the alkaline level of the solution is high, and there is no precipitation. When the evaporation of the solution was increased to 80%, precipitation occurred in the solution, and the precipitate was later examined. The solution with an evaporation rate of 75% was regarded as the optimal solution.

The determination of cations in the solution and precipitate and their precipitation was done by using flame photometry. Flame photometric analyses were performed using a BWB XP flame photometer produced by BWB Technologies Company (United Kingdom).

The determination of potassium ion concentration in the solution was done by taking 1.0 ml of the solution and placing it in a 1000 ml volumetric flask. Distilled water was added up to the mark, after which the solution was thoroughly mixed. Fifteen milliliters were withdrawn from the flask and analyzed using flame photometry.

Flame photometric analysis was done according to a calibration curve, and the concentration of the ions was determined in ppm.

The chemical composition of the precipitate was studied by the method of salt analysis. During the research work, a sample of the precipitate weighing 1 g was weighed exactly, placed in a volumetric flask of 1000 ml capacity, and then dissolved in distilled water. Then the mixture was well stirred, and the concentration of the main cations (potassium K^+, sodium Na^+, calcium Ca^{2+}, and barium Ba^{2+}) in the solution was determined using the flame photometry method.

The structural composition of the precipitate was established by infrared spectroscopy (FTIR). The measurements were carried out on an IRAffinity-1S FTIR spectrometer (Shimadzu, Japan). The spectra obtained allowed the identification of the functional groups present in the sample and an assessment of the mineralogical composition and chemical nature of the precipitate.

Results and Discussion

The research involved an analysis of the main stages of obtaining the alkali solution from cotton stalk ash, which is classified as agricultural waste, and the alkali concentration process. In order to

provide for complete dissolution of the alkaline components from the ash, the dissolution was carried out in water at a ratio of 1:5, providing for the creation of the primary alkali solution. The obtained solution was concentrated through gradual evaporation, thus creating solutions with varying degrees of concentration. In the present research, evaporation degrees of 25%, 50%, 75%, and 80% were tested, with the 75% degree being identified as the most technological one. The alkalinity was estimated by the titrimetric method, and the dependence on the evaporation degree was estimated. Additionally, the solutions with the 75% and 80% evaporation degrees, and the precipitate sample created under conditions of high concentration, were studied from the perspective of their physicochemical characteristics.

It was established that with an increase in the evaporation level, the balance of ions in the solution is disturbed, and individual substances pass into the phase of the precipitate in the form of insoluble compounds. In this regard, the impact on the selectivity of the composition of the solution, as well as the possibility of retaining the main alkaline components in the solution, is quite tangible.

According to the results of flame photometry of the optimal solution, potassium ions prevail in its composition. For a detailed evaluation of these data, the experimental results obtained during repeated tests are shown in Table 1.

Table 1 - Analytical results of the optimally evaporated solution

Experimental samples	Ion concentration (ppm)				
	K	Na	Ca	Ba	Li
Sample 1	114.9	10.1	9.3	8.5	0.35
Sample 2	115.3	10.3	9.5	8.7	0.38
Sample 3	116.1	10.6	9.8	9	0.42
Sample 4	116.5	10.8	10	9.2	0.45
Sample 5	115.7	10.2	9.4	8.6	0.4
SD	0.63	0.29	0.29	0.29	0.038

It is clear from the table above that the potassium ion had the highest concentration among all ions analyzed, and the low standard deviations suggest that the test results were highly reliable.

For instance, in the solution reduced to 75%, the mean concentration of potassium ions was 115.7 ppm, indicating that potassium is indeed the primary alkali element in the sample. However, apart from potassium ions, other ions such as Na^+, Ca^{2+}, and Ba^{2+} ions were also observed, albeit in low concentrations (Figure 1).

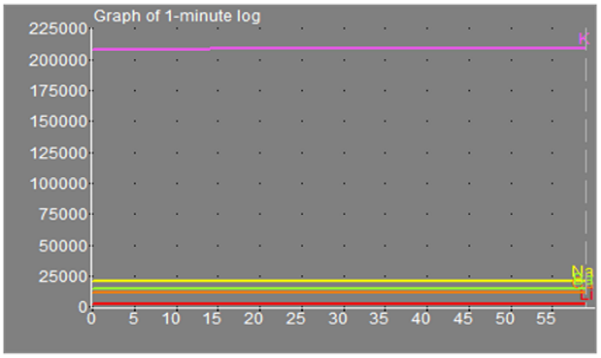

Figure 1 - Flame photometric spectrogram of the optimal solution

Based on the average values of the results from flame photometry, the presence of the following ions was detected in the solution: Na – 10.4 ppm; K – 115.7 ppm; Li – 0.4 ppm; Ca – 9.6 ppm; Ba – 8.8 ppm.

To express the values in terms of molar concentration, it was assumed that ppm is equal to mg/L, and then the calculations were carried out using the molecular mass of each ion.

Moreover, taking into account the fact that the solution sample was diluted 1000 times, the obtained values were recalculated using the appropriate coefficient, which allowed determining the concentration of ions in the initial solution (Figure 2).

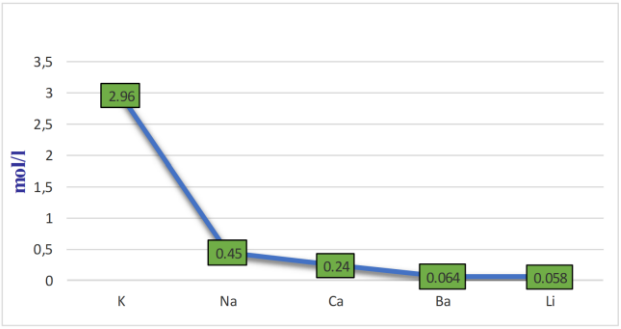

Figure 2 - Ion concentration in the optimal solution

Furthermore, considering that the solution was diluted up to 1000 times (from 1 to 1000 ml) during the test, it was taken into consideration that the resulting data referred to the sample diluted 1000 times, and accordingly, the resulting concentrations were recalculated by multiplying by 1000.

However, with the increasing evaporation degree, which means that the evaporation of the solution proceeded to more than 75%, the

precipitation took place. As shown in Table 2, based on the analysis of the precipitate carried out with the help of the BWB XP flame photometric technique, the precipitation includes not only Ca2+ ions but also considerable amounts of potassium ions.

Table 2 - Results of the analysis of the precipitate composition

Experimental samples	Ion concentration (ppm)				
	K	Na	Ca	Ba	Li
Sample 1	31.8	0.5	3.6	10.4	0.15
Sample 2	32.2	0.55	3.8	10.7	0.18
Sample 3	33	0.65	4.1	11.1	0.22
Sample 4	33.4	0.7	4.2	11.3	0.25
Sample 5	32.1	0.6	3.8	11	0.2
SD	0.67	0.07	0.24	0.35	0.038

From the table, it can be clearly seen that although different types of cations are found in the precipitate, the sum of all these fractions comprises a small proportion of the precipitate. The existence of potassium ions in the precipitate implies that evaporation of the solution should be stopped before the formation of the precipitate (up to 75% evaporation).

In addition, the existence of potassium ions in the precipitate suggests that potassium salts also partially precipitate from the solution at high concentrations.

The reason behind this behavior is the disturbance of the solubility equilibrium of the solution and the process of co-precipitation due to ion interactions. Meanwhile, the relatively large percentage of barium ions in the precipitate suggests the precipitation of carbonates, which means that carbonate ions are also present in the system and readily react with divalent cations to form poorly soluble compounds (Fig. 3).

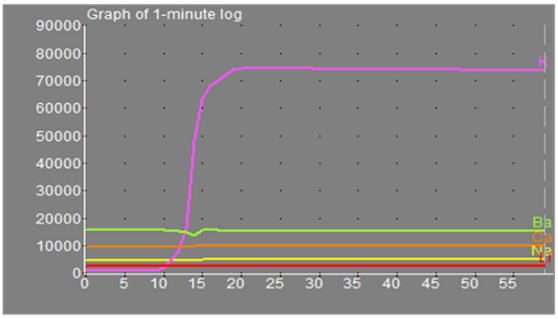

Figure 3 - Flame photometric spectrogram of the precipitate

Based on the average results of flame photometric analysis, it is possible to claim that the precipitate contains Na – 0.6 ppm, K – 32.5 ppm, Li – 0.2 ppm, Ca – 3.9 ppm, Ba – 10.9 ppm.

The analysis was performed according to the methodology, whereby 1 g of precipitate was used as a sample and was placed into a 1000 mL volumetric flask and filled up with distilled water. Then, a portion of this solution was analyzed with the help of a flame photometer. Consequently, the obtained ppm values indicate the content of ions in 1 g of precipitate in 1 L of solution.

The molar concentration of each ion was calculated, and the content of ions in the composition of the precipitate was estimated (Fig. 4).

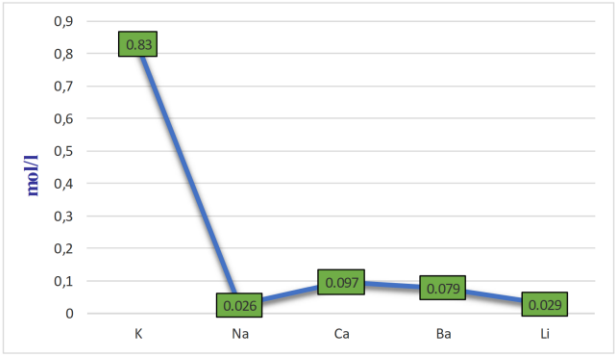

Figure 4 - Ion concentration in the precipitate

Based on the obtained data, it can be said that the content of potassium ions in the precipitate exceeds the content of other alkali metal ions. Thus, along with calcium, the precipitate contains potassium salts. It can also be noted that due to the higher content of barium ions, sodium, and lithium ions, the composition of the precipitate is a heterogeneous mineral mixture. In general, based on the obtained data, the precipitate after evaporation of up to 80% belongs to the complex multicomponent mixture containing a large amount of potassium and carbonate components.

Data on the chemical nature of the precipitate obtained by means of infrared spectroscopy (IRAffinity-1S FTIR) were additionally important. The analysis of the received spectrum (Fig. 5) confirms that the precipitate is predominantly carbonate.

The very strong absorptions appearing at 1477.68 and 1400.32 cm^{-1} represent the ν_3 vibrational transitions of the CO_3^{2-} ion. This high level of intensity and broadness implies that the carbonate species predominate. Furthermore, the absorption band appearing at 837.11 cm^{-1}

represents the v_2 transitions of the CO_3^{2-} ion; additionally, the absorption bands at 696.30 and 638.44 cm^{-1} correspond to the v_4 transitions. Notably, the absorption bands in the ~700 cm^{-1} region reveal the carbonate phases and could imply the existence of $CaCO_3$ or other carbonate forms.

In this context, the very intense absorption appearing at 1238.30 cm^{-1} indicates the existence of ion-ion interactions within the carbonate system or mixed carbonate phases. Additionally, the absorption band appearing at 1555.55 cm^{-1} might signify some other types of bonding interactions in the carbonate system or possibly the presence of organics.

The band located at 988.70 cm^{-1} signifies the presence of silicate compounds (Si-O bonds) within the precipitate sample; hence, the material does not appear to be an entirely carbonate compound but a mineral mixture. This may be attributed to the existence of silicates in the form of biomass ash contaminants. Weak peaks appearing in the high-frequency range (2081, 2144, 2206, and 2264 cm^{-1}) could be due to atmospheric CO_2 or gaseous compounds. The weak peaks found in the low-frequency range (605.65 and 449.41 cm^{-1}) represent metal-oxygen (M-O) bonds.

Generally, based on the FTIR analysis results, one may conclude that the predominant content of the precipitation is represented by carbonate compounds, primarily calcium carbonate. Nevertheless, due to the presence of certain additional peaks, it can be stated that the composition of the precipitate is not pure $CaCO_3$ but is a complex mixture of carbonates containing potassium, sodium, and other elements.

In this regard, based on the data obtained using infrared spectroscopy and flame photometry methods, one can conclude that the principal component of the precipitate is $CaCO_3$, but its composition is complex.

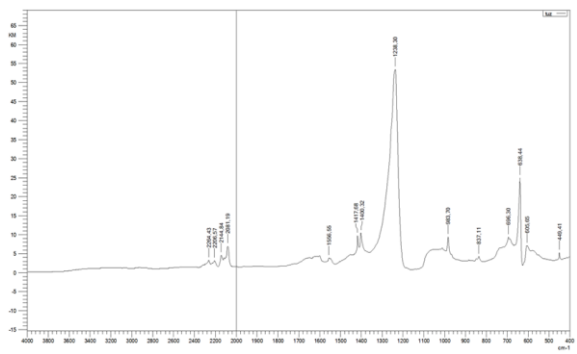

Figure 5 - FTIR spectrogram of the precipitate obtained using the IRAffinity-1S spectrometer

The obtained results indicated the distribution of ions between the solution and precipitate phases. At optimal parameters, potassium ions remained in the solution, while calcium ions migrated to the precipitate phase in the form of insoluble carbonates. Nonetheless, increasing the concentration of the solution resulted in disturbance of solubility equilibrium, thereby causing potassium compounds to migrate to the precipitate. The mechanism involved in this migration is called co-precipitation due to the ion interaction effect, which decreases the selectivity of the system.

It should be noted that the comparison of the results with literature data shows considerable discrepancies. Previously, it was found that potassium compounds remained in the solution phase, while in the current investigation, the migration of potassium ions occurred at a high degree of evaporation.

In this way, even though the evaporation procedure used for the concentration of the solution has positive effects up to a certain point, going past this point causes imbalance in the system and results in negative outcomes. According to the results of this experiment, it was concluded that the best technology in this situation is to stop the evaporation procedure before the appearance of precipitate. With such an approach, the largest amount of potassium ions will remain in the solution.

Conclusion

Based on the findings of this research, it became possible to define the basic physicochemical processes of extract solution evaporation from cotton stalk ash. It was established that the concentration of the solution up to 75% entails an increase in alkalinity, whereby potassium ions are present in a concentration of 2.96 mol/L, and precipitation does not take place. If the degree of evaporation exceeded 75%, then the solubility equilibrium would be disrupted, and precipitation would occur.

According to the analysis results, it is possible to conclude that the nature of the precipitation is predominantly carbonate, containing, besides other compounds, potassium ions and calcium. Thus, co-precipitation takes place owing to the interplay of ions when the concentration is high. Based on the analysis of ion distribution, it was revealed that potassium ions stay in the solution during ideal evaporation conditions, but excessive

concentration leads to their partial transition into the solid phase. This fact is accompanied by a decrease in selectivity and loss of useful substances. On this basis, it can be argued that from the technological perspective, the best regime implies the termination of the evaporation process prior to the appearance of precipitation, that is, at 75%.

According to the calculations, the total alkalinity of the obtained solution is 3.41 N due to K^+ and Na^+ ions. It is known that in conventional vegetable oil refining technology, sodium hydroxide (NaOH) is usually used as a 20% solution with a density of approximately 1.2 g/mL, which corresponds to about 6 N alkalinity for oil with an acid value of 5 mg KOH/g. The comparison results showed that the alkalinity of the ash extract solution is approximately 1.8 times lower than that of the same volume of NaOH solution used in conventional technology, meaning that its alkaline effect is about 56.8%.

However, from a technological point of view, the total amount of alkali introduced can be compensated by appropriately increasing the volume of the solution. On this basis, the use of ash extract solution may significantly reduce the consumption of imported NaOH and, under certain conditions, partially or almost completely replace it. The effectiveness of this approach in vegetable oil refining will be studied in detail in future research.

Conflicts of interest. On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement: **M. Boltayev and Q. Serkayev:** Conceptualization, Methodology, Software, Data curation, Writing draft preparation; **M. Boltayev, M. Khamidova and B. Khakimova:** Visualization, Investigation; **B. Khakimova:** Supervision; **G. Otajanova**: Software, Validation; **Akramova**: Reviewing and Editing.

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Қабылданды: *1 шілде 2026*

ТҮЙІНДЕМЕ

Бұл мақалада мақта сабағы күлінің судағы экстракты негізінде алынған сілтілі ерітіндінің оңтайлы буландыру режимі, сондай-ақ ерітіндіні буландыру барысында пайда болатын тұнбаның химиялық құрамына кешенді талдау жүргізілген. Мақта сабағының күлі тұрмыстық мақсаттарда, атап айтқанда нан мен самса пісіруге арналған тандыр пештерінде биомассаны жағу нәтижесінде түзілетін, қазіргі уақытқа дейін іс жүзінде пайдаланылмай, қоршаған ортаға шығарылып жатқан екінші реттік өнім ретінде қарастырылады. Ұсынылған зерттеуде сілтілі ерітіндіні алу үдерісі оңтайлы жағдайда жүргізілді, яғни күл мен судың 1:5 масса қатынасындағы қоспасы қайнатылды. Кейін алынған суспензия сүзгіден өткізіліп, буландыру жүргізілді. Алынған нәтижелер бойынша буландырудың оңтайлы режимі 75% деп анықталды, өйткені осы деңгейде тұнба түзілуі байқалмайды. Алайда буландыру дәрежесі 80%-ға жеткенде, ерігіштік тепе-теңдігінің бұзылуына байланысты ерітіндіде тұнба түзілуі анықталды. Тұнбаның катиондық құрамы жалынды фотометрия әдісімен анықталып, фазалық құрамы FTIR Фурье спектроскопиясы арқылы зерттелді. Нәтижесінде тұнбаның негізінен карбонат текті қосылыстардан тұратыны анықталды. Ерітіндіде калий иондарының басым екендігі және олардың молярлық концентрациясы басқа катиондарға қарағанда едәуір жоғары екендігі дәлелденді. Сонымен қатар, жоғары концентрация жағдайында калий иондарының белгілі бір бөлігі ерітіндіден тұнба фазасына өткендігі байқалды. Есептеулер бойынша алынған ерітіндінің жалпы сілтілігі 3,41 N- құрайды, бұл өсімдік майларын тазарту үдерісінде қолданылатын 6 N натрий гидроксиді (NaOH) ерітіндісінің сілтілігінің шамамен жартысына сәйкес келеді.

Түйін сөздер: мақта сабағының күлі, экстракция, буландыру, калий иондары, карбонаттар, тұнба, жалынды фотометрия, FTIR, бірлесіп тұнбалану, сілтілі ерітінді.

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Оптимальный режим выпаривания щелочного раствора, полученного из золы стеблей хлопчатника, и химический анализ процесса образования осадка

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АННОТАЦИЯ

В данной статье представлено комплексное исследование оптимального режима выпаривания щелочного раствора, полученного из водного экстракта золы стеблей хлопчатника, а также изучен химический состав осадка, образующегося в процессе выпаривания. Зола стеблей хлопчатника рассматривается как побочный продукт, образующийся при сжигании биомассы в бытовых устройствах, в частности в тандырах, используемых для выпечки хлеба и самсы, который в настоящее время практически не используется и выбрасывается в окружающую среду. В представленном исследовании процесс получения щелочного раствора осуществлялся в оптимальных условиях — путем кипячения смеси золы и воды при массовом соотношении 1:5. Затем полученная суспензия подвергалась фильтрации и выпариванию. По результатам установлено, что оптимальным режимом выпаривания является уровень 75%, поскольку при таком значении образование осадка не наблюдается. Однако при степени выпаривания 80% в растворе было зафиксировано образование осадка, что обусловлено нарушением равновесия растворимости. Состав осадка по катионам определялся методом пламенной фотометрии, а фазовый состав — методом ИК-Фурье спектроскопии (FTIR). Установлено, что осадок преимущественно состоит из соединений карбонатной природы. Показано, что в растворе преобладают ионы калия, при этом их молярная концентрация значительно превышает концентрации других катионов. Кроме того, при высокой степени концентрации часть ионов калия переходит из раствора в осадочную фазу. Согласно расчетам, общая щелочность полученного раствора составляет 3,41 N, что примерно соответствует половине щелочности 6 N раствора гидроксида натрия (NaOH), используемого в процессе рафинирования растительных масел.

Ключевые слова: зола стеблей хлопчатника, экстракция, выпаривание, ионы калия, карбонаты, осадок, пламенная фотометрия, FTIR, совместное осаждение, щелочной раствор.

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