

# OBTAINING COLLAGEN HYDROLYZATES FROM SECONDARY PRODUCTS OF CHROME-TANNED LEATHER

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## OBTAINING COLLAGEN HYDROLYZATES FROM SECONDARY PRODUCTS OF CHROME-TANNED LEATHER

**ABSTRACT.** In many tanneries, chrome-tanned leather shavings are still viewed as waste, despite their high collagen content and potential for reuse. This study focused on developing a process to extract collagen hydrolysates from these shavings using alkaline hydrolysis. The lab work involved several phases of testing with NaOH, KOH, and Ca(OH)<sub>2</sub> under different concentrations and temperatures, followed by further purification using barium chloride. The results of the study showed that chromium levels dropped dramatically, and the collagen that remained had strong adhesive properties, measured using standard surface tension and contact angle methods. Early experiments showed swelling issues and filtration delays, which the researchers had to troubleshoot by adjusting hydrolysis time and alkali ratios. But by the final stage, the method proved reliable. The broader takeaway here is that collagen recovery from chrome waste could not be just chemically viable—it could also offer real industrial and environmental value if adopted more widely.

**KEYWORDS:** adhesive properties, alkaline hydrolysis, chrome-tanned leather, collagen hydrolysates, waste recycling

## OBȚINEREA HIDROLIZATELOR DE COLAGEN DIN PRODUSE SECUNDARE ALE PIELII TĂBĂCITE ÎN CROM

**REZUMAT.** În multe tăbăcării, răzătura de piele tăbăcită în crom este considerată deșeu, în ciuda conținutului ridicat de colagen și a potențialului de reutilizare. Acest studiu s-a concentrat pe dezvoltarea unui proces de extragere a hidrolizatorilor de colagen din aceste răzături folosind hidroliza alcalină. Lucrările de laborator au implicat mai multe faze de testare cu NaOH, KOH și Ca(OH)<sub>2</sub> la diferite concentrații și temperaturi, urmate de o purificare ulterioară folosind clorură de bariu. Rezultatele studiului au arătat că nivelurile de crom au scăzut dramatic, iar colagenul rămas a avut proprietăți adezive puternice, măsurate folosind metode standard precum tensiunea superficială și unghiul de contact. Experimentele timpurii au indicat probleme legate de gonflare și întârzieri la filtrare, pe care cercetătorii au trebuit să le rezolve prin ajustarea timpului de hidroliză și a raporturilor alcaline. Dar, până în etapa finală, metoda s-a dovedit fiabilă. Concluzia generală este că recuperarea colagenului din deșeurile de piele tăbăcită în crom nu ar putea fi doar viabilă din punct de vedere chimic – ar putea oferi, de asemenea, o valoare industrială și de mediu reală dacă este adoptată pe scară mai largă.

**CUVINTE CHEIE:** proprietăți adezive, hidroliză alcalină, piele tăbăcită în crom, hidrolizate de colagen, reciclarea deșeurilor

## OBTENTION D'HYDROLYSATS DE COLLAGÈNE À PARTIR DE SOUS-PRODUITS DU CUIR TANNÉ AU CHROME

**RÉSUMÉ.** Dans de nombreuses tanneries, les copeaux de cuir tanné au chrome sont encore considérés comme des déchets, malgré leur teneur élevée en collagène et leur potentiel de réutilisation. Cette étude s'est concentrée sur le développement d'un procédé d'extraction d'hydrolysats de collagène à partir de ces copeaux par hydrolyse alcaline. Les travaux en laboratoire ont comporté plusieurs phases de tests avec NaOH, KOH et Ca(OH)<sub>2</sub> à différentes concentrations et températures, suivies d'une purification supplémentaire au chlorure de baryum. Les résultats ont montré une diminution drastique de la teneur en chrome et de fortes propriétés adhésives du collagène restant, mesurées par les méthodes classiques de tension superficielle et d'angle de contact. Les premières expériences ont révélé des problèmes de gonflement et des retards de filtration, que les chercheurs ont dû résoudre en ajustant la durée d'hydrolyse et les proportions d'alcali. Finalement, la méthode s'est avérée fiable. L'enseignement principal est que la récupération du collagène à partir des déchets de cuir tanné au chrome pourrait non seulement être chimiquement viable, mais aussi présenter un réel intérêt industriel et environnemental si elle était plus largement adoptée.

**MOTS-CLÉS :** propriétés adhésives, hydrolyse alcaline, cuir tanné au chrome, hydrolysats de collagène, recyclage des déchets

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## INTRODUCTION

The issue of rational waste utilization remains one of the most pressing global challenges. On the one hand, waste is a significant source of environmental pollution; on the other hand, it serves as a raw material for valuable products through recycling processes. For example, in the leather industry, waste generated during production and processing—as well as by-products from secondary manufacturing processes—can be considered recyclable raw materials [1].

Leather industry waste can be classified into chrome-free and chrome-containing leather waste, depending on the presence of chromium. The most hazardous type of waste pollutants are chrome-containing wastes. Chrome leather shavings typically contain about 90% collagen and 2–4% chromium [2]. Therefore, the efficient recycling of such waste is crucial for the sustainable development of the leather industry and can bring significant economic benefits. The most effective method for processing chrome leather shavings involves separating collagen and chromium, which can be done using high-level and low-level hydrolysis techniques [3].

Chrome leather shavings are generated during the leveling of leather surfaces. In the leather processing stage, uneven parts of the hide (such as the tail sections or parts of the head that are unsuitable for production) are removed. During the leveling of thick hides, up to 10% chrome leather shavings are produced. These shavings typically have an average width of about 10 mm, a length not exceeding 120–150 mm, and a thickness ranging from 0.5 to 1 mm. Chrome leather shavings fall under the category of secondary raw material waste from leather production [4].

Currently, unused portions of these shavings are often dumped in open areas near leather processing facilities, posing serious

environmental risks [5]. Waste containing chromium can cause respiratory problems and a decreased ability to fight various diseases [6]. Moreover, the incineration of such waste contributes to severe air pollution due to the release of toxic hexavalent chromium (Cr+6), halogenated organic compounds, aromatic hydrocarbons, and other pollutants [7].

This study aims to extract collagen hydrolysate from secondary chrome-containing waste in the leather industry, minimize the volume of waste, implement an environmentally safe processing method, and follow the principles of “green technology”—representing an innovative and sustainable approach in this field.

## EXPERIMENTAL

### Alkaline Hydrolysis Process

During the recycling of chrome leather shavings, approximately 60–75% of collagen (Cr<sub>2</sub>O<sub>3</sub>) remains bound within the chrome-containing waste. The separation and removal of the chromium element from chrome leather shavings—known as dechromation—are carried out through a hydrolysis process under both alkaline and acidic conditions. Alkaline hydrolysis enables the removal of chromium from solid leather waste without damaging the collagen matrix.

The high efficiency of chromium removal depends on three key factors: firstly, alkaline treatment effectively breaks the bonds between chromium and collagen, forming a precipitate of Cr(OH)<sub>3</sub>. Secondly, acid treatment dissolves the Cr(OH)<sub>3</sub> precipitate, thereby partially separating chromium from collagen. Thirdly, the complete separation of chromium and collagen can ultimately be achieved through sequential or combined treatments.

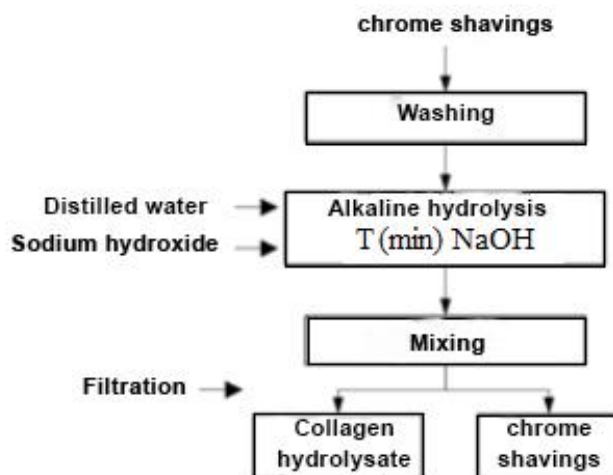


Figure 1. Alkaline hydrolysis process

A major drawback of the hydroxide-based method is that when acid is added to dissolve the  $\text{Cr}(\text{OH})_3$  precipitate, the collagen fibers—having lost their stabilizing chromium component—swell significantly. These swollen collagen fibers may restrict water release from the leather during filtration under pressure, thereby reducing process efficiency [8].

When peptide bonds in proteins are broken, the molecular chains are cleaved into two parts. Continued processing results in increasingly smaller fragments, making their removal easier. This progression not only facilitates more efficient chromium removal but also preserves the structural integrity of the leather [4].

The hydrolysis reaction of collagen proceeds as follows:



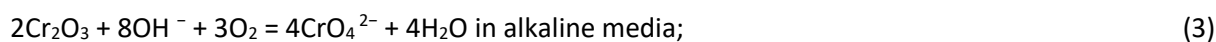
The chemical reaction for the chromium extraction process is usually as follows:



During the treatment of chrome shavings with alkaline solutions,  $\text{OH}^-$  ions displace the carboxyl groups in the collagen ion chains that are coordinated with  $\text{Cr}(\text{III})$ , leading to the formation of a  $\text{Cr}(\text{OH})_3$  precipitate and the release of  $\text{Cr}(\text{III})$  from the collagen fibers. However, separating  $\text{Cr}(\text{OH})_3$  from the collagen fibers remains a challenging task, as it requires the addition of acid to

dissolve and remove the precipitate. Preventing the re-binding of dissolved  $\text{Cr}(\text{III})$  and avoiding excessive hydrolysis of the collagen fibers are critical factors for achieving an efficient and complete separation of  $\text{Cr}(\text{III})$  from the collagen matrix.

The following reactions occur under alkaline conditions:



To address these issues, treatment with a solution of uniform concentration ( $\text{NaOH}$ , pH 13.5, 30 °C, for 3 hours, and 5%  $\text{Na}_2\text{SO}_4$ ) facilitates the formation of the chromium precipitate  $\text{Cr}(\text{OH})_3(\text{H}_2\text{PO}_4)_2^-$  through the hydrolysis of collagen fibers. The behavior of

chromium (III) under these conditions aligns with the reactions mentioned above [5].

Recycling chrome shavings significantly contributes to reducing pollution from the leather industry, while enabling the production of high-value functional materials. It has been scientifically proven that collagen

hydrolysate is an excellent adhesive, and chrome leather waste contains up to 17% adhesive content. Hydrolyzing chrome-containing leather waste not only enables the separation of chromium but also supports its application in product manufacturing—one of today’s most pressing environmental and industrial challenges.

## Materials and Methods

### *Process for Obtaining Collagen Hydrolysates from Chrome Leather Waste*

To extract collagen hydrolysates from chrome-containing leather waste, samples were collected from the “Yuksalish Charm Sanoat” LLC enterprise in the Namangan region and transported to the scientific laboratory of the “Chemical Technology” Department at the Namangan Institute of Engineering and Technology. Before beginning the experiments, the chromium content and overall composition of the chrome leather waste were analyzed under ISO 5398-1 standards. These tests were carried out using the EDX-7200 laboratory analyzer, and the results are presented in Table 1.

Table 1: The amount of Cr element in the process after hydrolysis

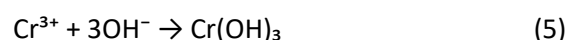
	Elements	Share (%)
1	Cr	53.533
2	Cl	29.524
3	S	12.665
4	Si	0.968
5	Ca	0.721
6	Fe	2.257
7	K	0.128
8	Zn	0.007

Based on the data obtained, it was found that the combined content of chromium (Cr) and chlorine (Cl) accounted for approximately 54%, significantly higher than other detected elements. To isolate chromium from the leather shavings, the alkaline hydrolysis method was applied. For this method, key reagents such as sodium hydroxide (NaOH), potassium hydroxide (KOH), and calcium hydroxide (Ca(OH)<sub>2</sub>) were used.

Hydroxide solutions were prepared at various concentrations, taking into account key parameters such as temperature and hydrolysis duration. These were then applied to selected ratios of chrome leather waste (based on dry mass), with a total reaction volume of 1 liter. The use of these alkaline agents facilitates the separation of chromium from the collagen matrix, precipitating it as chromium hydroxide [3].

After precipitation, the chromium hydroxide was dissolved using acid solutions and subsequently removed as a soluble salt via filtration [5].

In the first stage of hydrolysis, solutions of sodium hydroxide (NaOH), potassium hydroxide (KOH), and calcium hydroxide (Ca(OH)<sub>2</sub>) were used. The alkali loosens the collagen fibers, converting them into soluble peptides. For each 100 g sample, a 1:1.5 ratio of 5% KOH solution was heated at 70 °C for 20–60 minutes. Under the influence of temperature and the addition of chromium (Cr) with alkali (NaOH or KOH), a green precipitate—chromium(III) hydroxide (Cr(OH)<sub>3</sub>)—was formed:



When additional alkali was added to the chromium(III) hydroxide, the precipitate dissolved in the solution, and the green color disappeared, leaving the solution colorless. After the leather shavings were fully absorbed into the reaction, 3% NaOH was added, and continuous stirring was performed throughout the hydrolysis process. The results are presented in Table 2.

Table 2: The amount of Cr element in the process after hydrolysis

	Elements	Share (%)
1	K	0.294
2	Cr	0.235
3	S	0.054
4	Si	0.235
5	Ca	0.030
6	Fe	0.004
7	Cl	0.029
8	H <sub>2</sub> O	99.116

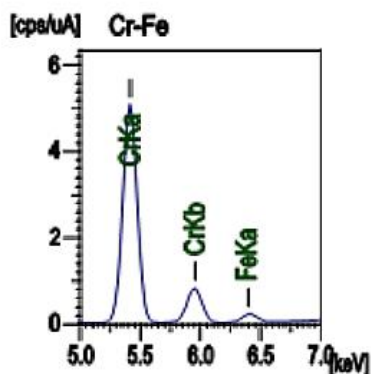


Figure 2. Results of X-ray spectral analysis of Cr and Fe elements

From the table, it is evident that after alkaline hydrolysis, the Cr content in chrome-containing leather waste decreased to 0.235%, indicating significant removal of chromium. After hydrolysis, the collagen hydrolysate was filtered and separated from the alkaline-treated chrome leather waste. The hydrolysate was stored in a measuring flask at 4 °C. Following the hydrolysis, the remaining Cr content in the chrome leather waste was re-analyzed using the EDX-7200 laboratory device (see Table 3).

Table 3: The amount of Cr in the filtered collagen hydrolysate

	Elements	Share (%)
1	K	0.282%
2	Cr	0.005%
3	S	0.102%
4	Si	0.248%
5	Cl	0.069%
6	Fe	0.000%
7	Zn	0.001%
8	H <sub>2</sub> O	99.289%

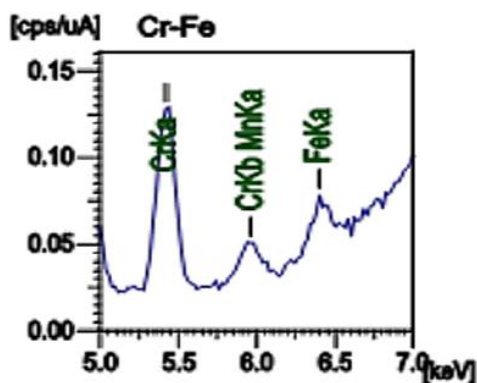
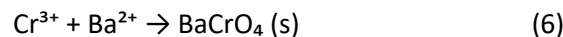


Figure 3. Results of X-ray spectral analysis of Cr and Fe elements

The filtered collagen hydrolysate was found to contain only 0.005% Cr, showing effective reduction. To completely remove Cr ions from the hydrolysate, a 5% BaCl<sub>2</sub> solution was added. As a result, a white precipitate of barium chromate (BaCrO<sub>4</sub>) was formed:



This reaction was used to detect the presence of chromium ions, as barium chromate is white and has very low solubility. The precipitate was then removed by filtration. To determine the final Cr content in the filtered collagen hydrolysate, a third-phase analysis was conducted, and the results are shown in Table 4.

Table 4: The amount of Cr element in the process after Ba hydrolysis

	Elements	Share (%)
1	Ba	2.175%
2	Cl	1.839%
3	S	0.016%
4	Si	0.237%
5	K	0.071%
6	Fe	0.000%
7	Zn	0.000%
8	H <sub>2</sub> O	95.550%

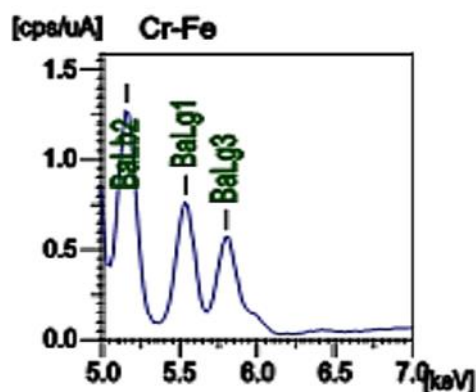


Figure 4. Results of X-ray spectral analysis of Cr and Fe elements

## RESULTS AND DISCUSSIONS

### Collagen Hydrolysate and Its Adhesive Properties

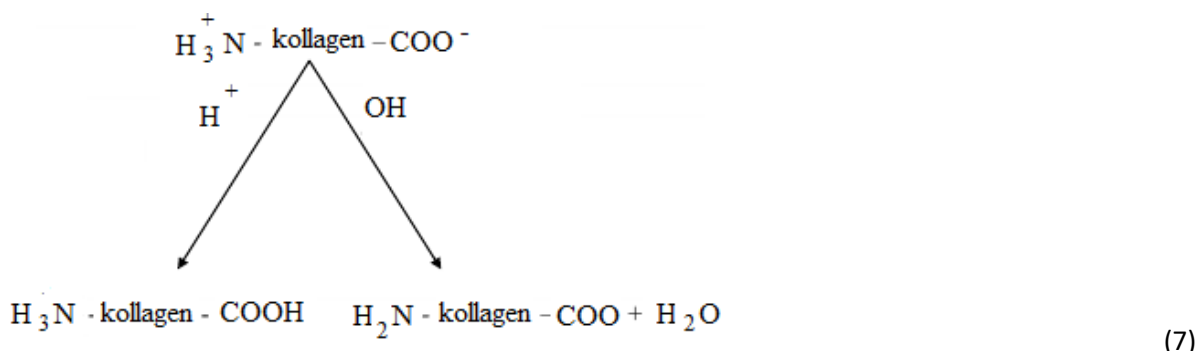
Analysis results confirmed that the collagen hydrolysates were completely purified from chromium. During alkaline hydrolysis, chromium(III) ions reacted with hydroxide ions, forming a Cr(OH)<sub>3</sub> precipitate,

while collagen was hydrolyzed. The effects of typical variables in the alkaline hydrolysis method—such as reaction time, alkali concentration (%), and temperature (°C)—on the degree of hydrolysis, total protein content, and its efficiency (%) were studied.

To this end, an optimization study was carried out using statistical experimental

models, and all obtained values were controlled under ISO 5398-1 standards.

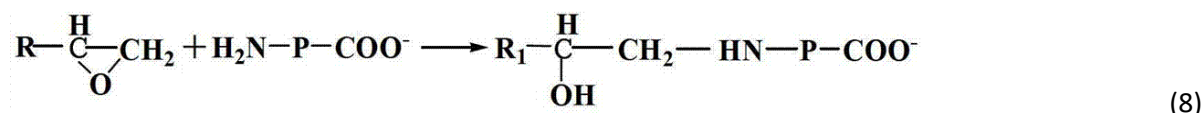
Based on the experiments conducted, the interaction of acid and alkaline agents with chrome-containing leather waste can be represented using the following conceptual process:



The main factors influencing the efficiency of collagen protein extraction from chrome leather waste include: type of alkali, amount of alkali used, primary hydrolysis temperature, water consumption, enzyme release and exposure time, and others.

From the above, it can be concluded that the method of recycling chromium shavings produced during the leather leveling process is characterized as a technical process

for extracting collagen protein, consisting of the following stages: Leather shavings → pre-treatment → primary hydrolysis → mixing → filtration of the solution → collagen hydrolyzes → amino acids and polypeptides are produced, and chromium precipitates. In the targeted alkaline hydrolysis method, the reaction for obtaining water-soluble collagen hydrolysate is as follows:



H<sub>2</sub>N – COO- structural formula of collagen hydrolysate:



In preliminary studies of alkaline hydrolysis, reaction time and NaOH concentration were identified as the key variables controlling the process. Using this method, collagen hydrolysate was successfully extracted from chrome-containing leather waste. The product obtained through high-temperature heating of collagen was referred to as “glue.”

**The adhesive properties**—Wa (work of adhesion), Y (surface tension), and the

equilibrium contact angle—were calculated according to GOST 10028-81 [9]. Contact angles were measured on glass slides using 5% collagen hydrolysate under a horizontal microscope. After introducing a droplet onto the gel surface and waiting 3 minutes, the measured angle was recorded as the equilibrium contact angle. Measurement accuracy was ±2°.

Table 5: Adhesion properties of collagen hydrolysates in different technological variants

Options	Temperature °C	Adhesion, N/m	Mass fraction, %
Process 1: Hydrolysis with NaOH	50	1598	0.20
Process 2: Hydrolysis with NaOH	70	1619	0.21
Process 3: Hydrolysis with NaOH	90	1658	0.22
Control	70	1588	0.28
According to GOST-325280	70	at least 1570	At most 0.3

The resulting collagen hydrolysate was water-soluble, and when heated above 90 °C, it formed a thicker, jelly-like gel. After heating, the hydrolysate was collected and its adhesive properties were studied. The results are summarized in Table 5.

As shown in the table, under hydrolysis conditions using NaOH at 90 °C, the collagen hydrolysate exhibited the highest adhesive strength of 1658 N/m. Across all experimental samples, the collagen hydrolysates obtained from chrome leather waste showed high adhesive performance. The final product was odorless, with a structure resembling sticky, adhesive gel.

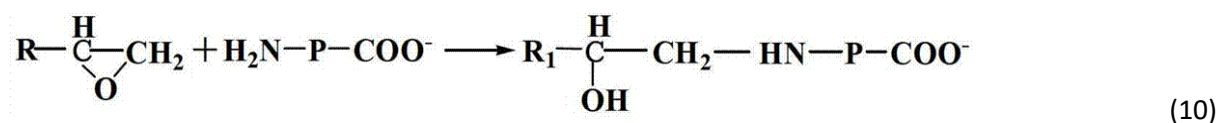
Additionally, the third experimental process demonstrated approximately 37% efficiency, which was higher compared to the first and second trials. The pH level of the collagen hydrolysates was found to be in the 7–8 range, indicating a neutral to mildly alkaline nature.

## CONCLUSIONS

During the process of obtaining collagen hydrolysates from chrome-containing leather waste, the content of chromium (Cr) and the total amount of other elements present in the waste were analyzed using the EDX-7200 laboratory analyzer. In alkaline hydrolysis, chromium(III) ions react with hydroxide ions, resulting in the formation of trivalent chromium hydroxide precipitate (Cr(OH)<sub>3</sub>), while the collagen undergoes hydrolysis.

The influence of key variables specific to the alkaline hydrolysis method—such as reaction time, alkali concentration (%), and temperature (°C)—was evaluated in terms of hydrolysis efficiency, total protein yield, and process effectiveness (%). Among them, reaction time and sodium hydroxide concentration were identified as critical parameters for process optimization.

As a result of the experiment, chromium present in the chrome shavings was successfully separated, and collagen hydrolysate was obtained.



Notably, under hydrolysis conditions using NaOH at 90 °C, the resulting collagen hydrolysate exhibited a high adhesive strength of 1658 N/m, indicating excellent adhesive properties.

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