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Article

Preliminary studies regarding the hydrolysis of leather waste in order to use as raw material in the fertilizer industry

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Abstract
Large amounts of tanned leather waste can be utilized as a valuable raw material in the fertilizer industry by embracing circular economy principles. Due to their high collagen content, which provides an essential nitrogen source (14%), these wastes hold significant potential. However, to incorporate them into fertilizer formulations, leather waste must first be hydrolysed to produce gelatine. This study focuses on developing an optimal method for hydrolysing tanned leather waste. The experiments investigated the effects of several parameters - including the type and concentration of hydrolysis agent, temperature, and contact time - on the degree of hydrolysis achieved. Leather waste samples were treated with various hydrolysis agents (water, sulfuric acid, oxalic acid, potassium hydroxide, and ash) at different concentrations and temperatures ranging from 40 °C to 110 °C for periods between 60 and 160 minutes. Results indicated that basic hydrolysis was the most effective approach. Treating leather waste with 0.5 M KOH at 100 °C for 120 minutes, with a stirring speed of 400 rpm and a 1:10 solid-to-liquid ratio, resulted in a degree of hydrolysis of 98.58%. Comparable results (97.25% hydrolysis) were obtained with a 1:2 ash-to-water leachate under similar conditions (100 °C for 100 minutes, 400 rpm, 1:10 ratio). These findings demonstrate that both potassium hydroxide and ash leachate are highly effective agents for the hydrolysis of tanned leather waste, supporting their integration into sustainable fertilizer production.

Keywords: *leather waste valorization, leather hydrolysis, alkaline hydrolysis, acid hydrolysis*

INTRODUCTION

The leather industry generates approximately 6 million tons of solid waste annually, which can be repurposed as a raw material for the fertilizer industry due to its high collagen content [1÷3]. Additionally, the significant nitrogen content in leather waste (up to 10%) enables its use as an alternative nitrogen source in the production of NPK fertilizers [4,5].
However, before leather waste can be utilized as a raw material in the fertilizer industry, it must first undergo preliminary dechroming (for chrome-tanned waste) and hydrolysis.
The hydrolysis step is essential, as it transforms solid leather waste into a liquid (gelatinous) phase [6÷8]. Leather hydrolysis is a chemical process in which the protein component of the skin (mainly collagen) is broken down into simpler substances, such as peptides and amino acids. The nature and extent of hydrolysis depend on the agents used (acids, bases, water, or enzymes) and the specific operating conditions. Hydrolysis can be partial or total [7,9,10]. During partial hydrolysis, only some peptide bonds in collagen are broken, resulting in the formation of gelatin [11,12]. In total hydrolysis, collagen is completely degraded into amino acids and small peptides, yielding a liquid product. Table 1 summarizes the main hydrolysis agents used in studies reported in the specialized literature.
Masilamani et al. conducted a comparative study on the hydrolysis of collagen from goat leather waste using two organic acids: propionic acid and acetic acid. Through repeated extraction processes

with 0.5 M propionic acid or 0.5 M acetic acid in the presence of 5% sodium chloride, they achieved hydrolyzed collagen yields of 93% for propionic acid and 85% for acetic acid [13].

Xu and colleagues reported the acid hydrolysis of leather waste using an inorganic acid, specifically 20% phosphoric acid [14]. By treating skin waste samples with phosphoric acid under vigorous magnetic stirring at 80°C for 2 hours, they obtained hydrolyzed collagen enriched with 6% phosphorus.

Table 1. Hydrolysis agents used in the hydrolysis studies

Hydrolysis agent used	References
Propionic acid Acetic acid	[13]
Phosphoric acid	[14]
Sulfuric acid Dipotassium phosphate trihydrate	[15]
Dipotassium phosphate trihydrate	[16,17]
Potassium hydroxide	[18]
Sodium hydroxide Potassium hydroxide	[19]

Similarly, Tzoumani and collaborators achieved an elastic, semi-transparent hydrolyzed collagen at 7% (w/v) through acid hydrolysis with inorganic sulfuric acid. Furthermore, phosphorus- and potassium-enriched hydrolyzed collagen was produced by reacting leather waste with 2÷3.5% sulfuric acid, 2.5÷3.5% dipotassium phosphate ($K_2HPO_4 \cdot 3H_2O$), and 0.5÷1% sodium chloride at 86÷90°C for 1.5÷2.5 hours in an autoclave [15].

The buffering capacity of dipotassium phosphate tetrahydrate has been leveraged in various hydrolysis studies. Constantinescu and collaborators described a direct method for hydrolyzing leather waste under alkaline conditions, resulting in a gelation product suitable for fertilizer applications [16]. Their process involved initial washing of the waste with water, followed by decalcification using ammonium sulfate, and then alkaline hydrolysis with 3÷4.5% dipotassium phosphate ($K_2HPO_4 \cdot 3H_2O$) at 75÷80°C for 1.5÷3.5 hours. Similarly, another reported approach used dipotassium phosphate to hydrolyze bovine leather waste. In this method, the waste was first washed with water and then treated with 5÷6.5% $K_2HPO_4 \cdot 3H_2O$ for 60÷120 minutes at 85÷96°C, resulting in a hydrogel [17].

Studies on alkaline hydrolysis have demonstrated the effectiveness of bases such as potassium hydroxide and sodium hydroxide in breaking down leather waste. For example, Hu and colleagues used 1 M potassium hydroxide (KOH) at 60°C for 1 hour, obtaining a potassium-enriched hydrolysate [18]. In another study, Gousterova and collaborators achieved basic hydrolysis of rabbit and sheep leather waste by treating the protein material with a mixed solution of 0.05 M NaOH and 0.15 M KOH for 12÷24 hours at 102 ±5°C [19].

Drawing on the existing research, the present study proposes a comparative analysis of the hydrolysis process for dechromed leather waste in three reaction environments: acidic, basic, and neutral.

MATERIALS AND METHOD

Materials

The sample used in the study was leather waste dechromed in six steps with water, oxalic acid, and ethylenediaminetetraacetic acid [20]. The leather waste sample was cut into small pieces (<1 cm) and dried at room temperature before hydrolysis. The content of total organic carbon and total nitrogen was determined using a CHNS Elemental Analyzer, while the content of potassium was determined using Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The amounts of total chromium and phosphorus were determined using Atomic Absorption Spectrometry (AAS). The physical-chemical characterization of dechromed leather waste is shown in Table 2.

All reagents used in this study - oxalic acid, sulfuric acid, and potassium hydroxide - were of analytical grade. The ash sample used in the experiments was derived from wood combustion and was physically and chemically characterized for moisture content, total organic carbon, total nitrogen, phosphorus, potassium, and chromium. The results are presented in Table 3.

Table 2. The physical-chemical characterization of leather waste sample

Parameter name	M.U.	Value	Methods applied
Water content	%	7.84	SR EN ISO 21660-3:2021-3
Dry substance	%	92.16	SR EN ISO 21660-3:2021-3
Total nitrogen	% d.m.	12.56	SR EN ISO 21663:2021
Total organic carbon	% d.m.	49.51	SR EN ISO 15936:2022
Total chromium	mg/kg d.m.	366.8	SR EN 16171:2017
Potassium	mg/kg d.m.	27.84	SR EN 16171:2017
Phosphorus	mg/kg d.m.	0.1336	SR EN 16171:2017

Note: d.m. dry matter

Table 3. The physical-chemical characterization of the ash

Parameter name	M.U.	Value	Methods applied
Water content	%	0.68	SR EN ISO 21660-3:2021-3
Dry substance	%	99.32	SR EN ISO 21660-3:2021-3
Total nitrogen	% d.m.	<0.1	SR EN ISO 21663:2021
Total organic carbon	% d.m.	7.99	SR EN ISO 15936:2022
Total chromium	mg/kg d.m.	12	SR EN 16171:2017
Potassium	mg/kg d.m.	150552	SR EN 16171:2017
Phosphorus	mg/kg d.m.	0.0623	SR EN 16171:2017

Note: d.m. dry matter

The hydrolysis process was based on the contact of dechromed leather waste with three different environments: acid conditions (sulfuric acid, oxalic acid), neutral conditions (water), and alkaline conditions (ash: water leachate, potassium hydroxide) to obtain the best hydrolysis yield. Also, the influence of some important parameters, such as: concentration of the hydrolysis agent (oxalic acid 6.5%÷12.5%; potassium hydroxide 0.1 M÷0.5M; ash: water leachate 1:2÷1:15), hydrolysis temperature (40ºC÷110ºC), and contact time (60÷160 min) was investigated.
The hydrolysis degree of the sample was calculated using the following equation:

$$Hyd.\ degree = (wa-wb)/wa \times 100, \quad (1)$$

where: *Hyd. degree* is the hydrolysis degree of the sample (%); *wa* is the mass of initial sample (g); *wb* is the mass of residue after hydrolysis (g).

Influence of hydrolysis agent
The samples were put into contact with five different hydrolysis agents: distilled water, 0.1 M potassium hydroxide, ash–water leachate (1:7), 3% sulfuric acid, and 6.5% oxalic acid. Hydrolysis was carried out at 40°C for 60 minutes, with a stirring speed of 400 rpm and a solid-to-liquid ratio of 1:10. After the reaction time, the samples were filtered to assess the degree of hydrolysis.

Influence of extraction agent concentration
The samples were contacted with varying concentrations of hydrolysing agents: potassium hydroxide (0.1M÷0.5M), ash-water leachate (ratios from 1:2 to 1:15), and oxalic acid (6.5%÷12.5%). The experiments were conducted at 40°C for 60 minutes, with a stirring speed of 400 rpm and a solid-to-liquid ratio of 1:10. After the contact time, the mixtures were filtered to assess the degree of hydrolysis.

Influence of temperature
The samples were put into contact with 0.5 M potassium hydroxide at various temperatures ranging from 40°C to 110°C, using a solid-to-water leachate ratio of 1:2. The mixture was stirred at 400 rpm for 60 minutes. After the contact period, the samples were filtered to determine the degree of hydrolysis.
Influence of contact time
The samples were put into contact with hydrolysis agents - 0.5 M potassium hydroxide and ash-water leachate at a 1:2 ratio - for varying contact times ranging from 60 to 160 minutes. The experiments were conducted at 100°C, with a stirring speed of 400 rpm and a solid-to-liquid ratio of 1:10. After the specified contact time, the mixtures were filtered to assess the degree of hydrolysis.

RESULTS AND DISCUSSION
Sample characterization
Chemical characterization of the sample revealed a low chromium content (366.83 mg/kg dry substance), supporting its suitability for use in the fertiliser industry. Additionally, the high nitrogen content (12.56% dry substance) in the leather waste makes it a promising alternative nitrogen source for smart fertilisers. However, analyses showed low levels of two essential macronutrients, phosphorus and potassium, in the skin sample. The analysed skin waste contained 0.1336 mg/kg dry substance of phosphorus and 27.84 mg/kg dry substance of potassium.

Hydrolysis agents' effect
The studies on the influence of the hydrolysis agent type on the degree of hydrolysis of the sample (Fig. 1) demonstrated that the hydrolysis environment directly influences the final result.

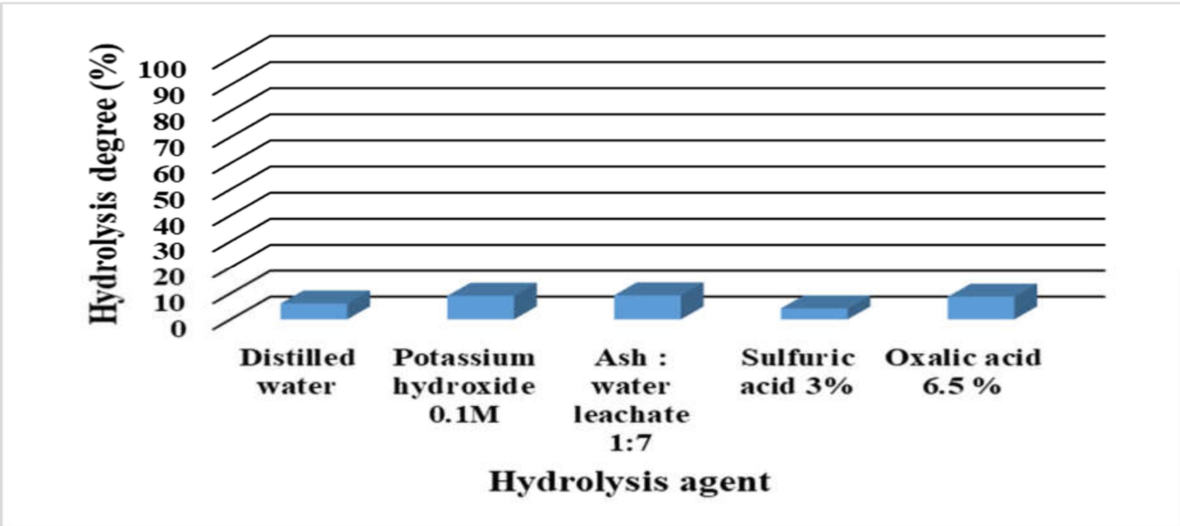

Fig. 1. The influence of hydrolysis agent type on the hydrolysis degree

The studies on acid hydrolysis showed that hydrolysis is more significant with the use of an organic acid (oxalic acid, 6.5%), with a degree of hydrolysis of 8.71%. When the sample was contacted with inorganic acid (3% sulfuric acid), only 4.23% of the sample was hydrolysed. *The hydrolysis experiments revealed that distilled water acts as a moderate hydrolysis agent, achieving 6.12% hydrolysis across the tested media.* On the other hand, studies on alkaline hydrolysis with 0.1 M potassium hydroxide have demonstrated the highest degrees of hydrolysis in ash: water leachate. Following contact with 0.1 M potassium hydroxide, a degree of hydrolysis of 9.05% was obtained, while upon contact with leachate ash: water 1:7, a degree of hydrolysis of 9.25% was obtained. The appearance of the samples after hydrolysis with different hydrolysis agents is shown in Fig. 2.

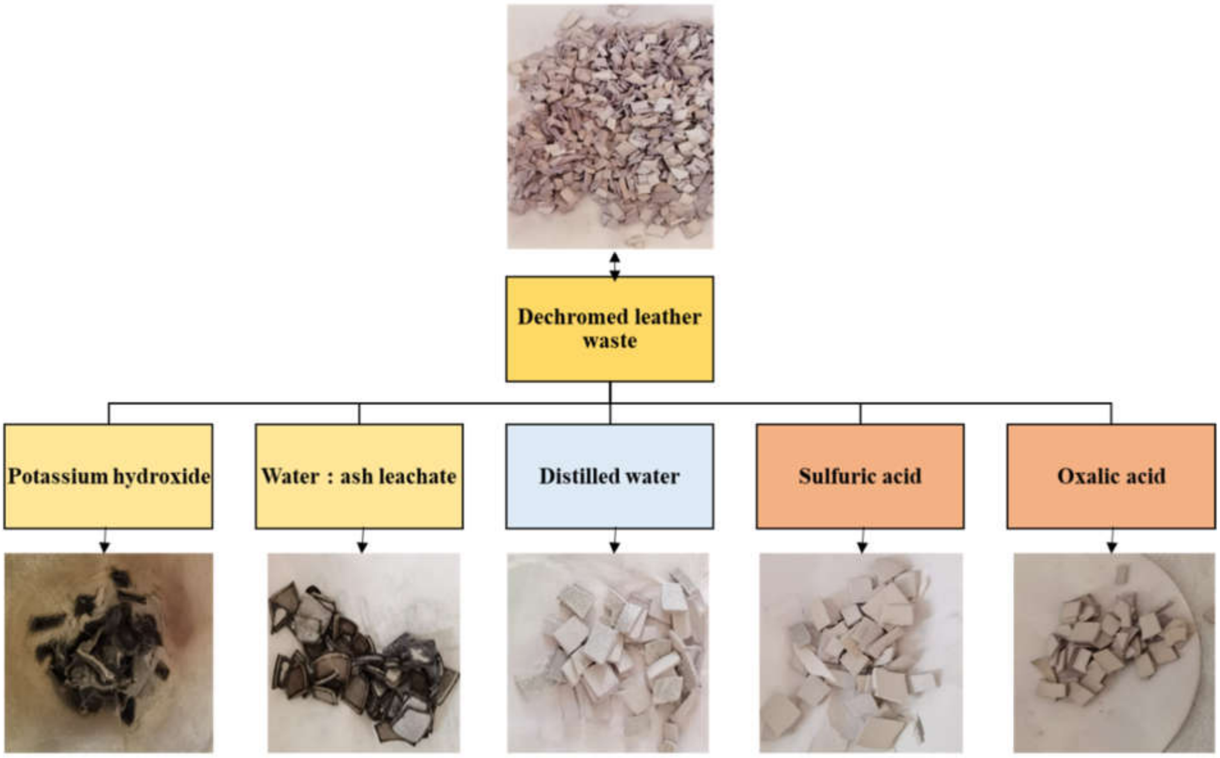

Fig. 2. The appearance of the samples after the hydrolysis process

At the end of the studies on the influence of the type of hydrolysis agent, it was concluded that potassium hydroxide, ash: water leachate, and oxalic acid are the optimal hydrolysis agents and will be tested in subsequent steps.

Oxalic acid concentration effect

The studies carried out on the influence of oxalic acid concentration (6.5%- 13%) on the degree of hydrolysis of the leather waste (Fig. 3, Fig. 4) demonstrated a low efficiency of oxalic acid in the hydrolysis process.

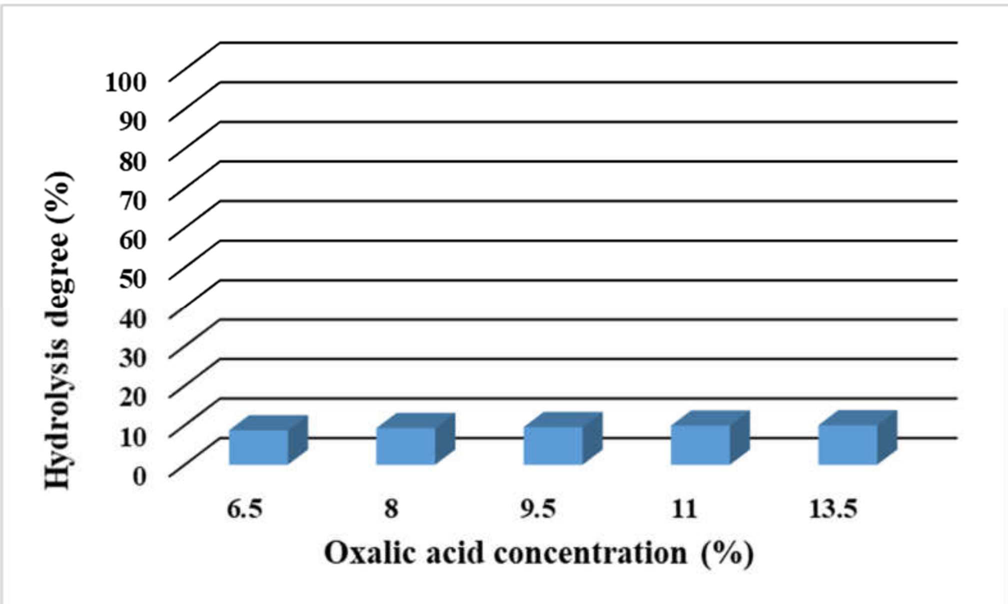

Fig. 3. The influence of oxalic acid concentration on the hydrolysis degree

The experimental results revealed that, in the concentration range studied (6.5÷13%), only a small fraction (<10%) of the skin waste sample hydrolyzes.

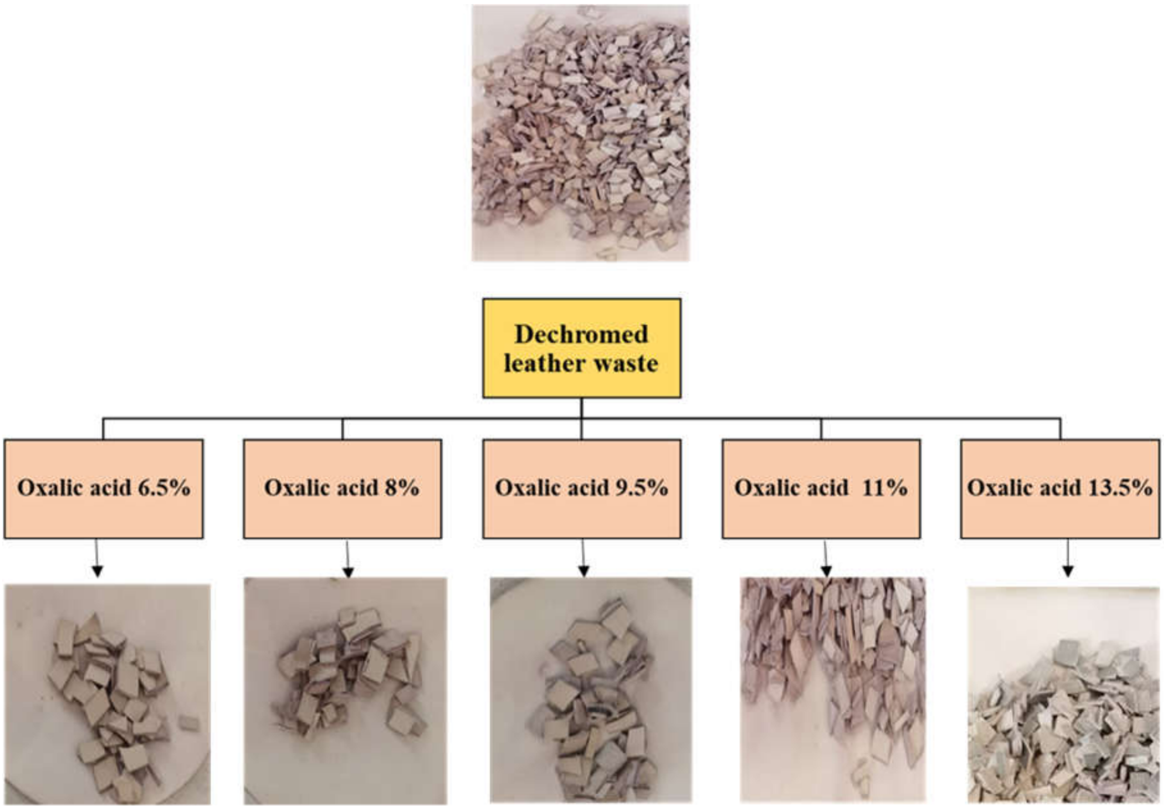

Fig. 4. The appearance of the samples after the hydrolysis process with different concentrations of oxalic acid

Potassium hydroxide concentration effect

The experimental studies on the influence of potassium hydroxide concentration on the hydrolysis of leather waste (Fig. 5) have highlighted a direct, proportional relationship between the base concentration and the degree of hydrolysis of the waste.

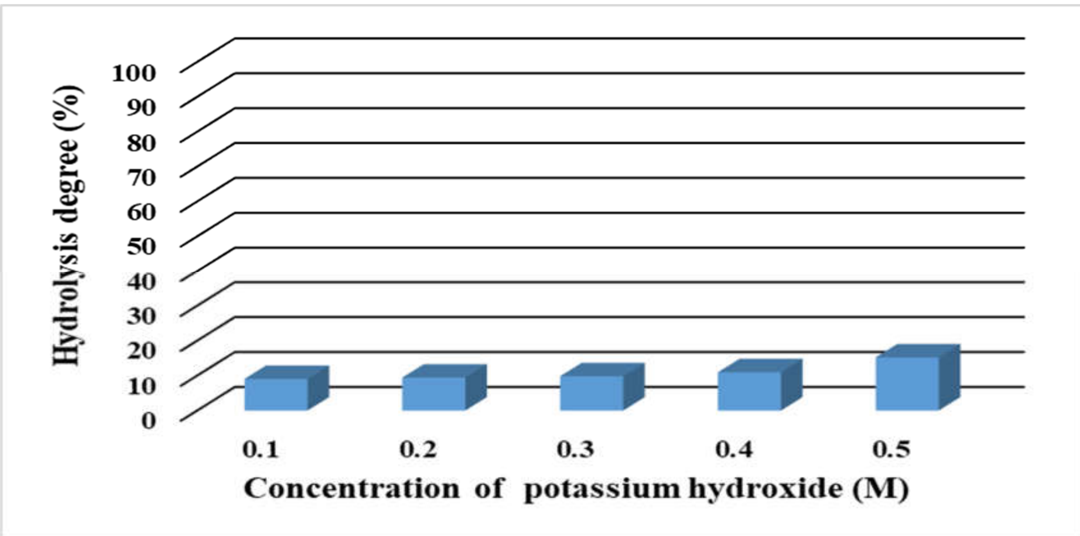

Fig. 5. The influence of potassium hydroxide concentration on the hydrolysis degree

Thus, in the potassium hydroxide concentration range studied (0.1÷0.5M), an increase in the degree of hydrolysis of the collagenous waste was observed when the potassium hydroxide concentration increased. When contacting the collagenous waste with 0.1 M KOH, the lowest result (Hyd. degree 9.05%) was obtained. At the opposite end, contacting the sample with 0.5 M potassium hydroxide yielded the highest degree of hydrolysis (15.22%). The appearance of the samples after hydrolysis with different concentrations of potassium hydroxide is shown in Fig. 6.

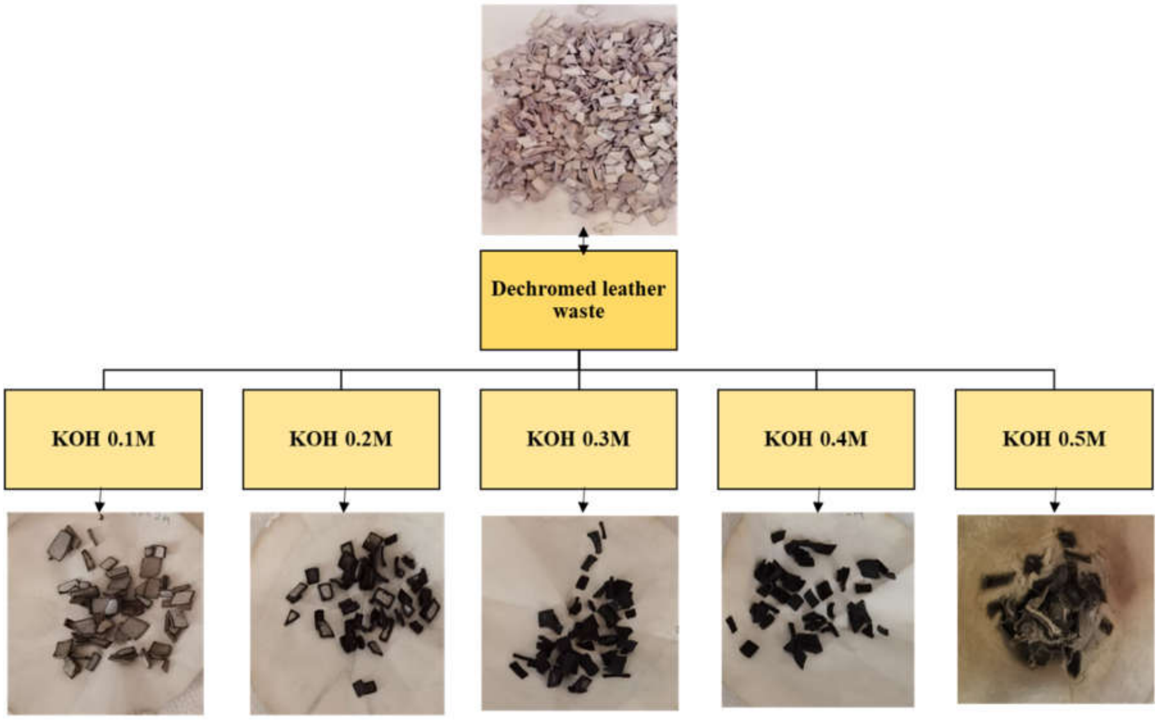

Fig. 6. The appearance of the samples after the hydrolysis process with different concentrations of potassium hydroxide

Ash: water leachate ratio effect

The results for the sample's contact with the ash-water leachate (Fig. 7) showed that, as the ash: water ratio increased, the degree of hydrolysis decreased.

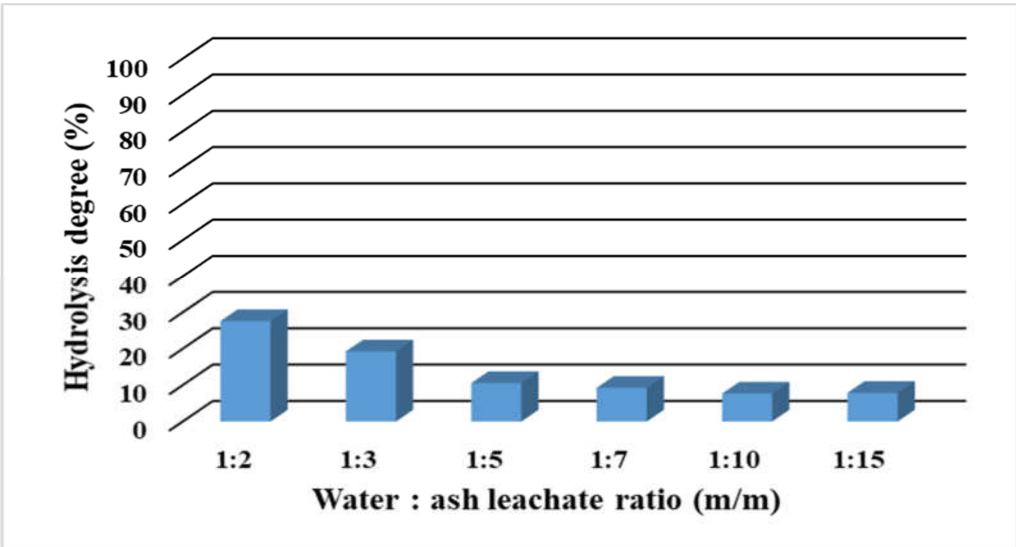

Fig. 7. The influence of ash: water leachate ratio on the hydrolysis degree.

It was observed that the leather waste sample placed in contact with a basic leaching solution prepared by mixing one part of ash with two parts of water undergoes the most significant hydrolysis (degree of hydrolysis 27.59%). At the opposite pole regarding the hydrolysis capacity of leather waste was the slightly basic solution obtained after leaching one part of ash with 15 parts of water (degree of hydrolysis 7.88%). The appearance of the samples after hydrolysis at different ash: water ratios is shown in Fig. 8.

At the end of the studies on the influence of hydrolysis agent concentration, it was concluded that potassium hydroxide 0.5 M and ash: water leachate 1:2 are the optimal hydrolysis agents.

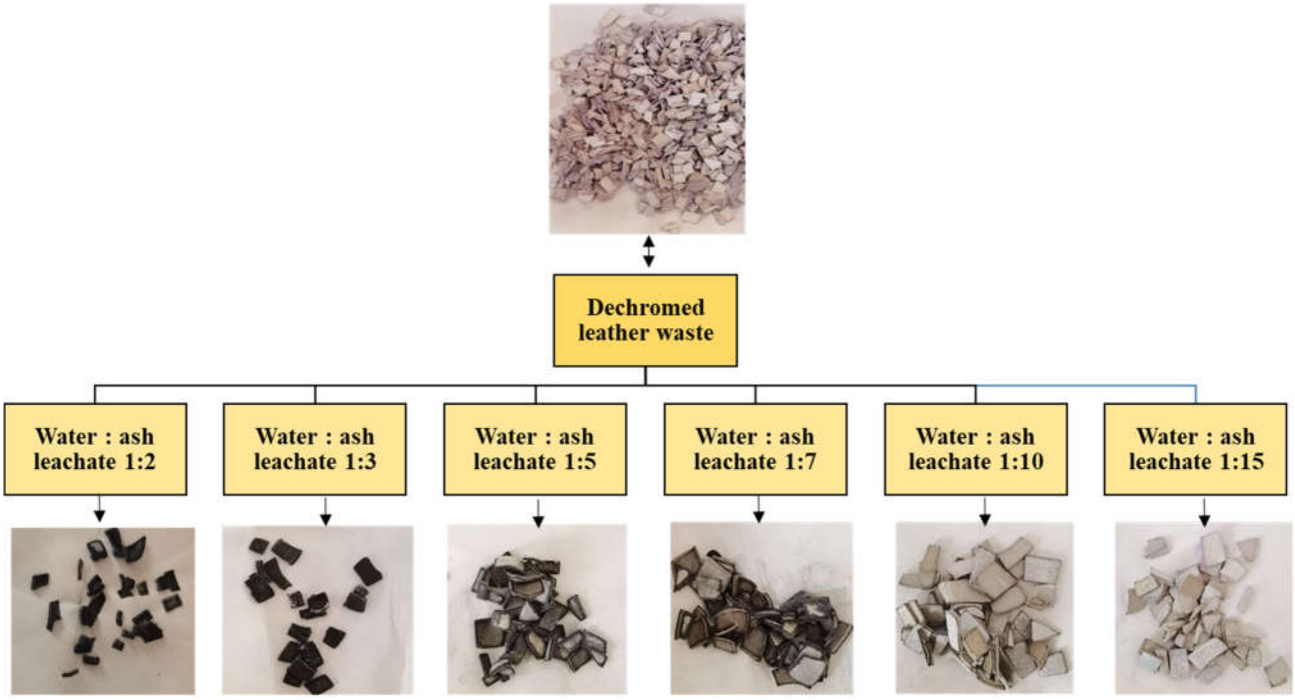

Fig. 8. The appearance of the samples after the hydrolysis process with different ash: water ratio

Temperature effect

The studies regarding the influence of temperature on the degree of hydrolysis were based on contacting a dechromed leather waste sample with 0.5 M potassium hydroxide, respectively with an ash: water leachate 1:2, at different temperatures (40÷110ºC). Experimental studies have shown that temperature is a key factor in the hydrolysis of dechromed leather waste.

By contacting the waste sample with a 0.5 M potassium hydroxide solution (Fig. 9, Fig. 10), it was observed that, in the temperature range 40÷60ºC, a partial hydrolysis of the sample occurs. The degree of hydrolysis obtained in this interval varied from 15.22% to 42.68%. Instead, as can be seen in Fig. 9, in the temperature range 70÷110ºC, the degree of hydrolysis of the waste increases rapidly from 84.23% to 96.10%. The best degree of hydrolysis was obtained at 110ºC (96.10%), but the value is close to that at 100ºC (96.00%).

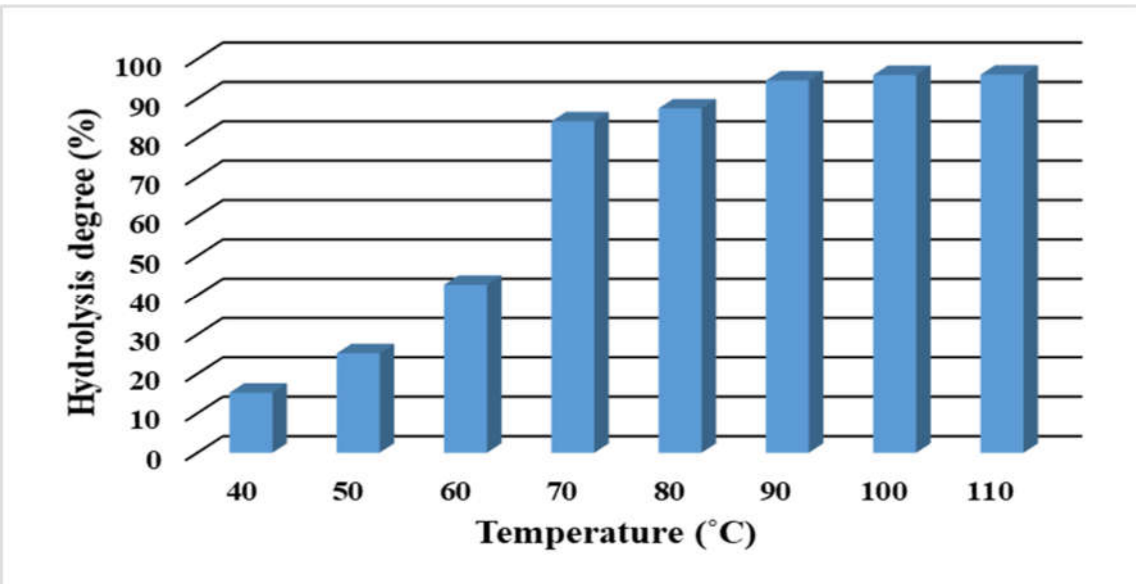

Fig. 9. The influence of temperature on the hydrolysis degree for samples treated with potassium hydroxide 0.5M

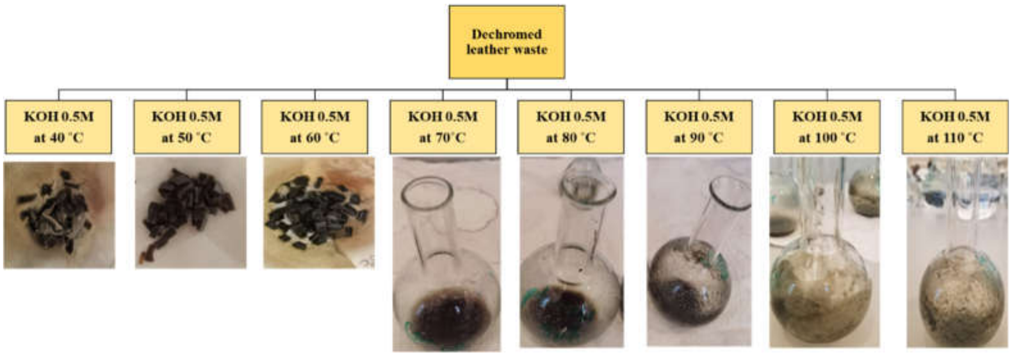

Fig. 10. The appearance of the samples after the hydrolysis process with potassium hydroxide 0.5M at different temperatures

At the same time, by contacting the sample with a basic ash: water leach solution at 1:2 (Fig. 11, Fig. 12), a direct, proportional relationship between the applied temperature and the degree of hydrolysis was observed. In the temperature range 40÷70ºC, partial hydrolysis of the sample occurs, with a degree of hydrolysis ranging from 29.59% to 67.86%. Instead, as can be seen in Fig. 11, in the temperature range of 80÷100ºC, the waste hydrolyses almost completely. The highest degree of hydrolysis was obtained at 100ºC (91.2%).

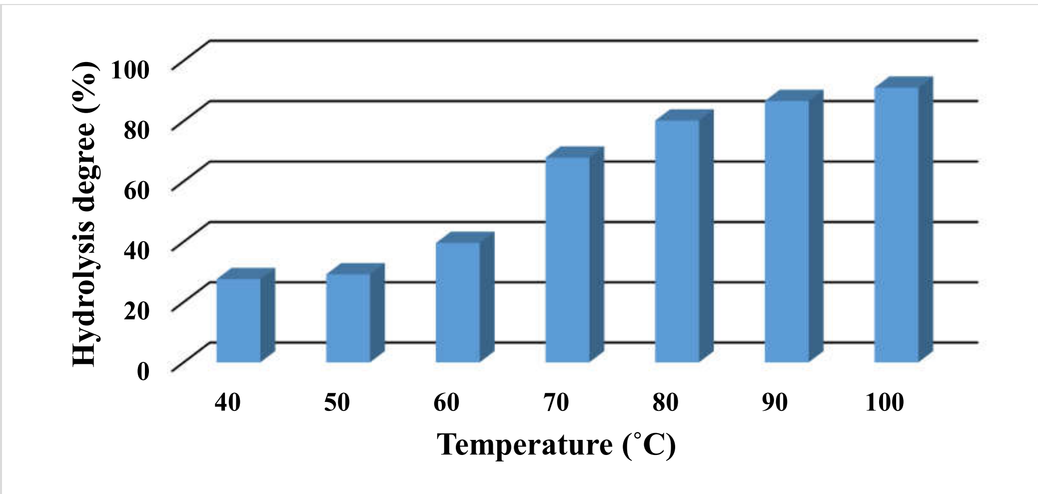

Fig. 11. The influence of temperature on the hydrolysis degree for samples treated with ash: water leachate 1:2

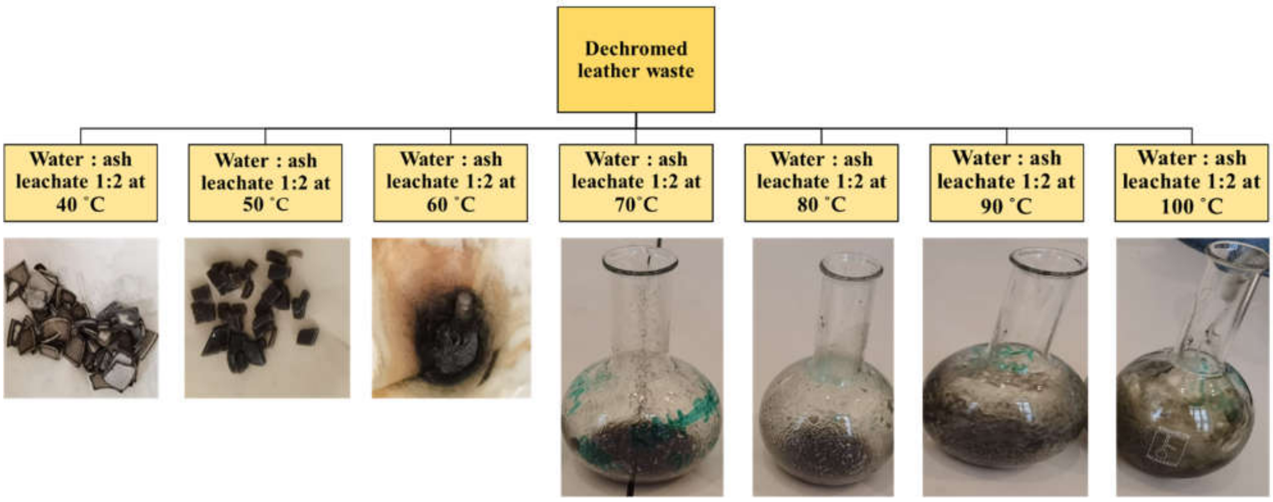

Fig. 12. The appearance of the samples after the hydrolysis process with ash: water leachate 1:2 at different temperatures

Contact time effect
By contacting the waste sample with potassium hydroxide 0.5 M (Fig. 13) for different periods of time (60÷160 min), it was possible to observe that the degree of hydrolysis improves slightly, increasing by approximately 2.7% in the studied interval. The highest degree of hydrolysis was obtained after 160 minutes of contact time (98.74%), a value close to that obtained after 120 minutes (98.58%) and 140 minutes (98.72%). At the end of the studies, it was concluded that contact time has a weak influence on the degree of hydrolysis. If you want to save time, the optimal contact time between the sample and 0.5 M KOH is 120 minutes (degree of hydrolysis 98.58%).

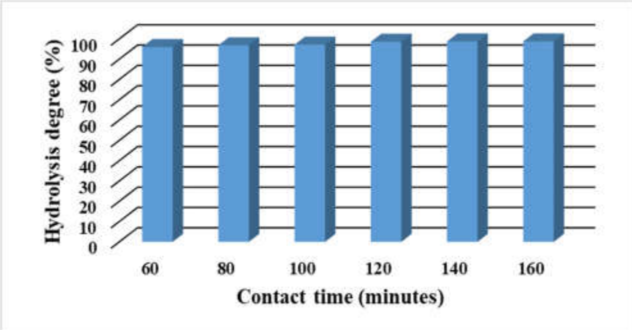

Fig. 13. The influence of contact time on the hydrolysis degree for samples treated with potassium hydroxide 0.5M

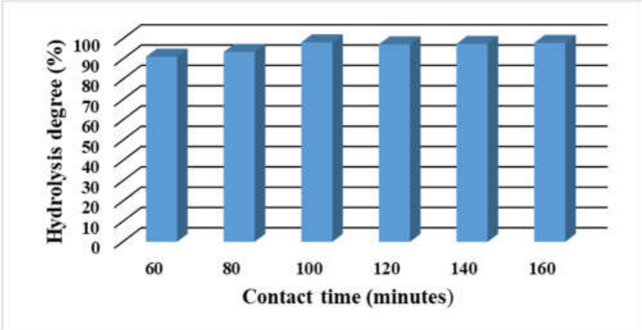

Fig. 14. The influence of contact time on the hydrolysis degree for samples treated with ash: water leachate 1:2.

At the same time, by contacting the sample with a basic ash: water leachate solution at 1:2 (Fig. 14), a direct, proportional relationship between contact time and degree of hydrolysis was observed. In the 60-100 minutes interval, an increase in the waste's hydrolysis degree from 91.20% to 97.25% was observed. However, between 100 and 160 minutes, no substantial improvement was recorded, with only a marginal increase of approximately 0.7% (from 97.25% to 97.99%). Based on these results, the optimal contact time between the sample and the ash-to-water leachate solution 1:2 was determined to be 100 minutes.

CONCLUSIONS

Hydrolysis of leather waste is a critical step in its valorisation as a raw material for the fertilizer industry. This study conducted parallel investigations of the hydrolysis process under alkaline, neutral, and acidic conditions. The results demonstrated that an alkaline environment is most effective for hydrolysing the studied waste. Additionally, experimental parameters such as the concentration of hydrolysis agents, temperature, and contact time were found to significantly influence the overall hydrolysis. Specifically, a hydrolysis yield of 97.25% was achieved by treating the sample with ash-water leachate (1:2 ratio) at 100°C for 100 minutes with stirring at 400 rpm. Furthermore, a degree of hydrolysis of 98.58% was obtained when the waste was treated with 0.5 M potassium hydroxide for 120 minutes at 100°C and 400 rpm.

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