Title: A PROCESS FOR COMPLETE UTILIZATION OF RAW HIDE / SKIN TO YIELD VALUABLE PRODUCTS

Abstract: The present invention provides a process for complete utilization of proteinaceous products from the raw hide/skin trimmings, wherein gelatin, protein hydrolysate containing mainly collagen and keratin hydrolysate are obtained. The process has potential applications in the manufacturing of pharmaceutical/food grade gelatin, collagen hydrolysates which can be used for food, pharmaceutical or agricultural applications, and keratin hydrolysate which has the potential for leather filler or other applications such as fertilizer, cosmetics.
A PROCESS FOR COMPLETE UTILIZATION OF RAW HIDE / SKIN TO YIELD
VALUABLE PRODUCTS

FIELD OF THE INVENTION

The present invention relates to a process for complete utilization of raw hide and/or skin to yield valuable products. In particular, the present invention relates to a process for the treatment of raw hide/skin to ensure their high value utilization. More particularly, the present invention relates to a process for the utilization of raw hide/skin to prepare gelatin, protein (collagen) hydrolysate and keratin hydrolysate. The process has potential applications in the manufacturing of pharmaceutical/food grade gelatin, collagen hydrolysates which can be used for varied applications from food, pharmaceuticals to agriculture, and keratin hydrolysate which has the potential for leather filler, fertilizer and other applications.

BACKGROUND OF THE INVENTION AND DESCRIPTION OF PRIOR ART

Leather manufacture is considered to be one of the major polluting sectors which generate huge amount of wastewater and solid waste like raw trimmings, fleshings, shavings, buffing dust and hair waste. Raw trimmings are proteinaceous solid waste generated prior to the pre-tanning processes in tannery. Trimming is the first unit operation carried out on the raw hides/skins, to trim off the edges viz., shank tail and head portions, in order to make the hides/skins amenable for mechanical operations during the subsequent stages of leather manufacture. For every metric ton of skin/hide processed about 50 to 70 kgs of raw hide/skin trimmings are generated, which is 5 to 7% of the weight of raw materials (predominantly wet salted hides/skins) processed for leather manufacture. Raw hide/skin trimmings are rich in collagenous protein and are not contaminated by any other chemicals. In current practices particularly in India, raw trimmings are either disposed of in landfills or used for preparation of low value products such as glue. In some countries the hide or skin is not completed recovered for leather manufacture and in such instances the opportunity for utilising the proteinous materials are completely missed. The technology covered under this patent provides newer opportunities for effective utilization of proteinous materials from the hides and skins. Some of the prior art disclosed here includes the sequential pre-treatment process for converting of solid wastes of leather industries into valuable products.

Reference may be made to US 20040030102A1 wherein disclosed is the process for gelatin extraction and chromium salt recovery from the tanned hide and skin shavings. The claimed
process consists of acid hydrolysis followed by separation of gelatin, chromium and hydrolyzing agent. The tanned hides/skin shaving was hydrolysed using 10-80% (w/w) of organic acids at 50-100°C. The solution was filtered and dialyzed with 1000-30000Da porous membrane for separating the gelatin and trivalent chromium. The solution was desalinated by ion exchange filter and diafilter using 200 to 500Da porous membranes for recovering the total chromium and hydrolyzing agent.

Reference may be made to CN101186786 B which describes the method for extracting unmodified natural collagen from the trimming wastes. They have claimed extraction of undenatured collagen, 100 parts by weight of raw bovine trimming was minced and pretreated with an alkali solution (pH 8-12) to make the skin swell for 24-48 hrs. Then it was bleached with 15-60 g/L of H₂O₂ at the pH of 8-12 for 3-8 hrs and was further demineralized with 150-250 parts by weight of 1.0 M EDTA or 0.2-1M HCl solution for 24-48 hrs. It was washed with an adequate amount of water and filtered to obtain sediment. For obtaining the native undenatured collagen solution, 100 parts of the sediment was treated with 0.5-6 parts of the protease at the pH of 2 to 4 at 6-8 °C for 1-3 days. Then the solution was centrifuged (16000-20000 rpm) to remove residues. The supernatant was added with 0.1-3M NaCl for salt precipitation. After precipitation, the precipitate was resuspended in 0.1-2 M acid. These steps were repeated 1-3 times and dialysed to obtain 5-8% of pure undenatured natural cow hide collagen solution.

Reference may be made to WO2007017402 A1 wherein disclosed is a process for obtaining protein hydrolysate in composition with manganese salt from the by-products of animal origin or leather tanning industry residues (before and after tanning process). The process comprised the step of hydrolysis by the action of base (lime) or acid (Sulphuric acid) or enzyme (proteolytic enzyme). After hydrolysis, calcium hydroxide and one or more manganese salts were added to the hydrolysed solution to promote the exchange reaction between the amino acid and peptides with calcium and manganese salt at 120°C, 2.0 bar for 1 hr. Excess calcium was precipitated out using precipitating agent (e.g., ammonium bicarbonate, sodium bicarbonate, oxalic acid). Then it was subjected to filtration and concentration to obtain protein hydrolysate with molecular weight of < 2000 Da.

Reference may be made to EP2284177 A1 which relates to preparing protein filler, for the leather industry, using animal hair obtained from un-hairing process of leather manufacture.
Hair was rinsed with tap water and soaked in 0.01-0.5 M of inorganic acid (e.g., Hydrochloric acid, sulfuric acid) for 10-48 hrs, and then neutralised with tap water, filtered and air dried. The dried hair (600 parts) was submerged with 2-30 wt% of reducing agent (e.g., Sodium mercaptoacetate, mercaptoethanol) for 10-48 hrs followed by filtering off the water and drying to obtain hair. The pretreated hair was hydrolysed by adding 0.5-20 wt% of alkaline compound (e.g., NaOH, KOH, Na<sub>2</sub>CO<sub>3</sub> etc.) at 50-95°C for 2-20 hrs to obtain thick liquid. After hydrolysis, the liquid was allowed to cool and then its pH was adjusted to 5-8 with an inorganic acid and filtered to obtain 10-20% of keratin polypeptide solution. The filtrate was concentrated and dried to obtain protein filler for making leather.

Reference may be made to RU2490286C1 wherein developed is a two step process to produce ecologically pure protein hydrolysate from the raw waste of leather industry. In a first step, raw waste is defatted using extraction solvent selected from the groups of ester at the reflux temperature (e.g. methyl acetate, ethyl acetate) of 50-125°C for 3-5 hrs, after which defatted residues were dried. In the second step, dried defatted residues were treated with unsaturated water vapor in soxhlet apparatus at a pressure of 1 atm and a temperature of 100°C for 5 hrs to obtain collagen/protein hydrolysate.

However, the major limitations associated with the processes available in the prior art may be summarized as follows:

- Most of the prior arts stated above towards the utilization of solid wastes from tannery involve a series of several sequential processes and involve the recovery of only any one of the proteinaceous products such as collagen/gelatin, protein hydrolysate, and keratin hydrolysate.

- In the case of trimming wastes only collagen protein is being utilized, moreover the same would involve a series of processes such as liming, dehairing, deliming and subjecting the trimming for treatment with a series of chemicals for preparing the waste for extraction of proteinaceous materials.

- The major limitations of these methodologies are the lengthy and time consuming pretreatment processes with numerous chemicals for recovering specific proteinaceous product.
• Also, purification methods like salt (e.g., NaCl) precipitation, dialysis and ion exchange filtration are involved in the recovery of final products.

• The prior art are time consuming and do not focus on maximizing the utilization of proteinous materials available in the trimming waste.

Thus, keeping in view the drawbacks of the hitherto reported prior art the inventors of the present invention realized that there exists a dire need to overcome the aforesaid shortcomings of the prior art and provide a simple and effective process for complete utilization of raw hides and skin trimming so as to yield three valuable products namely gelatin, protein (collagen) hydrolysate and keratin hydrolysate within 48 hrs.

OBJECTIVES OF THE INVENTION

The main objective of the present invention is to provide a process for the treatment of raw hide/skin after trimming, such that it results in complete utilization of raw hide and skin while resulting in valuable products which obviates the limitations of prior art.

Another objective of the present invention is to provide a process for extraction of the gelatin from the raw hide/skin trimmings prior to removal of hair.

Yet another object of the present invention is to extract the protein (collagen) hydrolysate after extraction of gelatin.

Yet another objective of the present invention is to make collagen hydrolysate directly from the hides and skins.

Still another objective is to provide a process for extracting the keratin hydrolysate from hair.

Yet another objective of the invention is to provide a simple process within short time period to recover (complete utilization) proteinous products from raw hide/skin trimmings.

SUMMARY OF THE INVENTION

The present invention provides a process for making three products viz., high grade gelatin,
protein (collagen) hydrolysate and keratin hydrolysate resulting in generation of high value products from raw hide/skin trimming. The process results in complete utilization of raw hide/skin trimming. Process associated with the extraction of gelatin prior to the removal of hair is a novel step compared to the conventional process steps which has a sequence of soaking followed by the removal of hair by liming process and hydrolyzing the un-haired hide/skin matrix for extraction of gelatin. Further, the conventional process is time consuming mainly due to the long liming process whereas the new process excludes all such time consuming process and all the three products are obtained within 48 hours. The developed process has potential applications in the manufacturing of pharmaceutical/food grade gelatin, protein hydrolysate (potential application in food, pharmaceutical and agriculture), and Keratin hydrolysate (potential application in the leather manufacturing as syntan, fertilizer, cosmetics, pharmaceutical and other applications). Hence the present invention provides a great scope for high value realization from waste.

In an embodiment, the present invention provides a process for the treatment of raw hide/skin trimming, which comprises:

[i] treating raw hide/skin after trimming with 5 to 90% (w/w) organic acid (% based on the weight of trimmings) at a temperature in the range of 45-70 °C for a period in the range of 15 to 24 hrs followed by separation of the resulting solution and subsequent drying at a temperature in the range of 4 to 70 °C to obtain gelatin,

[ii] hydrolysing the residual skin matrix, as obtained after separation in step [i], or raw hide/skin trimming preheated at the temperature of 60-80 °C, with 0.1 to 10% (w/w) of enzyme at the pH of 2.5-10 in presence of 100-300% (w/v) of water at 35 to 60 °C for period in the range of 2 to 10 hrs followed by treatment, as an optional step, with 2-15% (w/w) of caustic alkali, in presence of 100-300% of water at 1-5 atmospheric pressure and subsequent separation of the resulting solution and drying at a temperature in the range of 45 to 150 °C to obtain protein hydrolysate

[iii] treating the residual hair, as obtained after separation in step [ii], with 2-15% (w/v) of caustic alkali, in presence of 50-150% of water, at 90 to 120°C for period in the range of 3 to 12 hrs under 1-5 atmospheric pressure followed by separation
of the resulting solution and subsequent drying at a temperature in the range of 45 to 150 °C to obtain keratin hydrolysate.

In another embodiment of the present invention, protein source used may raw hides/skins of buffalo, cow, goat and sheep.

In another embodiment of the present invention, the organic acid used in step (i) for extraction of gelatin may be such as acetic acid, propionic acid.

In still another embodiment of the present invention, the enzyme for preparation of collagen hydrolysate used in step (ii) may be such as acid protease, neutral protease, alkali protease.

In yet another embodiment of the present invention, the separation method may be filtration, centrifugation.

In still another embodiment of the present invention, the pore size for separation may be in the range of 0.8 to 2 microns.

DETAILED DESCRIPTION OF THE INVENTION

The present invention provides a process for complete utilization of raw hide/skin generated from the tanning industries/slaughter houses so as to develop valuable products while reducing the environment concerns. The extraction of gelatin prior to the removal of hair is an important step as reported and claimed in the present invention as compared to the conventional process which has a sequence of soaking followed by the removal of hair by liming process and hydrolyzing the un-hairied hide/skin matrix for extraction of valuable products. More particularly in future, this invention shall create a massive impact on reducing the cost for preparing products such as gelatin, protein (collagen) hydrolysate and keratin hydrolysate.

Raw hide/skin and their trimmings are tannery/slaughter house by-products were used for the purposes of the present invention for extraction of high value products. Common name: Goat and scientific name: *Capra aegagrus hircus*.

Common name: Cow and scientific name: *Bos taurus*. 
Common name: **Sheep** and scientific name: *Ovis aries*.

Common name: **buffalo** and scientific name: *Bubalus bubalis*.

The Cow, buffalo, goat, sheep trimmings were collected from CLRI Pilot tannery, Chennai – 600 020, Tamil Nadu, India.

Acid and Neutral Proteases were procured from M/s Meteoric Exim Pvt Ltd (currently changed to M/s Meteoric Biopharmaceuticals Pvt Ltd), 2nd floor, Shiromani Complex, Opposite Ocean Park, Nr. Nehru Nagar cross Road, Ambawadi, Ahmedabad - 380015. Alkali protease was procured from M/s Tex Biosciences Pvt Ltd, Textan House, 75, Fourth Avenue, Ashok Nagar, Chennai - 600083.

Raw hide/skin trimmings were treated with 5 to 90% (w/w) of an organic acid (% based on the weight of trimmings) at a temperature in the range of 45-70 °C for a period of 15 to 24 hrs. The resulting solution was subjected to separation and the separated liquid was dried at a temperature in the range of 4 to 70 °C to obtain pure gelatin.

The residual skin matrix of the trimming, as obtained after the separation or raw hide/skin are preheated at the temperature of 60-80 °C and hydrolysed by treating with 0.1 to 10% (w/w) of protease at a pH of 2.5-10 in the presence of 100-300% (w/v) of water at 35 to 45 °C for period of 2 to 10 hrs. The reaction mixture was optionally treated with 2-15% (w/w) of caustic alkali, in the presence of 100-300% of water at 1-5 atmospheric pressure. The resulting solution was subjected to separation. The liquid was then dried at a temperature in the range of 45 to 150 °C to obtain protein hydrolysate.

The residual hair, as obtained after separation of the skin matrix, was treated with 2-15 % (w/v) of caustic alkali, in presence of 50-150% of water, at 90 to 120°C for period of 3 to 12 hrs under 1- 5 atmospheric pressure. The resulting keratin hydrolysate solution was subjected to separation and finally dried at a temperature in the range of 45 to 150 °C to obtain solid keratin hydrolysate.

A comparative overview of the conventional process for treating raw hide/skin trimming vis-
à-vis that of the present invention is presented in Figure 1, wherein the red line indicates the conventional process, whereas the green line indicates the process flow of the present invention. The inventive step of the present invention lies in the extraction of gelatin prior to the removal of hair, whereby it is possible to utilize both skin matrix and hair to obtain gelatin, protein hydrolysate and keratin hydrolysate.

**EXAMPLES**

*The invention is described in detail in the following examples, which are provided by way of illustration only and therefore should not be construed to limit the scope of the present invention.*

**Example 1**

One Kg of bovine raw trimmings were washed thrice with 3 lit of water to remove dirt and salt for 3 hrs. 1 kg of ethanol was mixed with 1 kg of hexane in a container and the mixture was acidified with 30g of acetic acid, whereby the pH was observed as 3.8. The washed trimmings were pre-treated with 2 kgs of this acidified hexane-ethanol mixture for 12 hrs and washed thrice with 2.5 lit of water for 2.5 hrs. The washed trimmings were then loaded into reactor and 2.5 lit of 4.5 % v/v acetic acid was added to the reactor. Temperature was maintained at 60°C. After a period of 3 hrs, 7 lit of 4.5 % v/v acetic acid was added continuously to the reactor at a constant flow rate of 8 mL/min. The reaction displaced gelatin solution from the reactor for a period of 15hrs. The temperature of the extraction was maintained at 60°C. During the extraction process, 4% of fat removal was achieved. The obtained gelatin solution was purified using 5 micron pre-filter followed by ultra-filtration using ceramic filter of size 0.8 micron, concentrated using nano filter of size 400 Da and then dried. Then the left over solution and residual trimming matrix with hair were enzymatically hydrolysed using 18 g of acid protease enzyme at 37.5°C for 6 hours to obtain protein hydrolysate. Followed by the recovery of protein hydrolysate, residual hair was treated with 6.5 g of NaOH with addition of 1 lit of water at 120°C with 15 psi for 4 hrs to obtain keratin hydrolysate. Yield of extracted gelatin was determined using hydroxyproline estimation and the physicochemical properties such as gel strength, viscosity were characterized. Yield and properties of protein, keratin hydrolysates were determined. Here, the yields of the three products were estimated on the basis of wet weight of the raw trimmings. Yield of the gelatin was ~18% and other properties viz., gel strength and viscosity are ~245 blooms and 3.5 cP respectively. Similarly, yield and physicochemical properties of the protein hydrolysate and
keratin hydrolysate were assessed. Yield of protein hydrolysate was ~12% of the weight of raw trimmings and as the molecular weight <5kDa. The yield of keratin hydrolysate was ~3.7% with a molecular weight <400Da.

Example 2
One Kg of bovine raw trimmings were washed thrice with 5 lit water to remove dirt and salt for 3 hrs. 1 kg of ethanol was mixed with 1 kg of hexane in a container and the mixture was acidified with 30g of acetic acid, whereby the pH was observed as 3.8. The washed trimmings were pre-treated with 2 kgs of this acidified hexane-ethanol mixture for 12 hrs and washed thrice with 2.5 lit of water for 2.5 hrs. The washed trimmings were then loaded into reactor and 2.5 lit of 5.5 % v/v propionic acid was added to the reactor. Temperature was maintained at 65°C. After a period of 3 hrs, 7 lit of 5.5% v/v propionic acid was added continuously to the reactor at constant flow rate 8mL/min. The reaction displaced gelatin solution from the reactor for period of 15hrs. The temperature of extraction was maintained at 65°C. During the extraction of process 3.5% of fat removal was achieved. The obtained gelatin solution is pre-filtered with 5 micron cartridge and purified by ultra-filtration using ceramic filter of size 0.8 micron and concentrated with nano filtration of size 400 Da and then dried. The left over hair with skin matrix was enzymatically hydrolysed using 20 g acid protease enzyme at ~37.5°C for 6 hours to obtain protein hydrolysate. After recovering of protein hydrolysate, 0.75 lit of water added with residual hair and treated with 7.5 g of NaOH at 120°C with 15 psi for 3.5 hrs to obtain keratin hydrolysate. Yield of extracted gelatin was determined using hydroxyproline estimation and the physicochemical properties such as gel strength, viscosity were characterized. Similarly, the yield and properties of protein, keratin hydrolysates were determined. Here, the yields of the three products were estimated on the basis of wet weight of the raw trimmings. Yield of the gelatin was ~16.8% and other properties such as gel strength and viscosity are ~255 blooms and 5.5 cP, respectively. Similarly, yield and physicochemical properties of the protein hydrolysate and keratin hydrolysate were assessed. Yield of protein hydrolysate was ~13.8% of wet weight of skin and has the molecular weight <6kDa. The yield of keratin hydrolysate was ~3.7% with a molecular weight <370 Da.

Example 3
One Kg of bovine raw trimmings were washed thrice with 4 lit of water to remove dirt and salt for 2.5 hrs. The washed trimmings were pre-treated with 40 g of lipase and 10 g protease
(w/w) for 3 hrs and are washed thrice with 2.5 lit of water. The washed trimmings were loaded into reactor and treated with 2.5 lit of 4.5 % (v/v) acetic acid to the reactor and temperature was maintained at 60°C. After the period of 2 hrs, 8 lit (w/v) of 4.5 % of acetic acid was added continuously to the reactor at constant flow rate of 7mL/min and the reaction displaced gelatin solution from the reactor for a period of 17 hrs. The temperature of extraction was maintained at 60°C. The obtained gelatin solution is purified by ultra-filtration using ceramic filter of size 0.8 micron, concentrated with nano filter of size 400 Da and then dried. The left over skin with hair matrix was enzymatically hydrolysed using 30 g of acid protease enzyme at ~38°C for 6 hrs to obtain protein hydrolysate. Followed by the recovery of protein hydrolysate, 0.75 lit of water added to the residual hair and treated with 8 g of NaOH at 120°C with 15 psi for 2 hrs to obtain keratin hydrolysate. Yield of extracted gelatin was determined using hydroxyproline estimation and the physicochemical properties such as gel strength, viscosity were characterized. Similarly, the yield and properties of protein, keratin hydrolysates were determined. Here, the yields of the three products are estimated on the basis of wet weight of the raw trimmings. Yield of the gelatin was ~16.5% and other properties such as gel strength and viscosity were ~220 blooms and 3.2 cP. Similarly, yield and physicochemical properties of the protein hydrolysate and keratin hydrolysate were assessed. Yield of protein hydrolysate was ~12.3% of wet weight of skin and has the molecular weight <6kDa. The yield of keratin hydrolysate was ~4.2% with a molecular weight <340Da.

Example 4

One kg of wet salted raw trimmings of goat skin were cut into pieces, washed with 2.5 lit of water to remove dirt and salt. Further, trimmings were pre-treated with 2.5 lit of 5% \( \text{H}_2\text{O}_2 \) (v/v) and washed twice with 3 lit of water. Then the washed trimmings were loaded into reactor and treated with 2.5 lit of 4.5% (v/v) of acetic acid at 55 °C. After a period 4 hrs, 6.5 lit of 4.5 % v/v acetic acid was added continuously at constant flow rate 10 mL/min. The reaction displaced gelatin solution from the reactor for a period of 18 hrs. The temperature of the extraction was maintained at 55°C and around 3.5% of fat removal was achieved. The obtained gelatin solution was purified by ultra-filtration using ceramic filter of size 0.8 micron and concentrated with nano filter of size 400 Da and then dried. Then the left over solution with hair and skin matrix were enzymatically hydrolysed using 24 g acid protease at 38°C for 4.5 hrs and filtered to obtain protein hydrolysate. After recovering the protein hydrolysate, residual hair added with 0.5 lit of water and treated at 120°C for 5.5 hrs at the
pressure of 15 psi and with 5g of NaOH. Yield of extracted gelatin was determined using hydroxyproline estimation and the physicochemical properties such as Gel strength, Viscosity were characterized. Yield of the gelatin was ~15.8% of wet weight of skin with gel strength of ~260 blooms and viscosity of 5.8 cP. Similarly, yield and physicochemical properties of the protein hydrolysate and keratin hydrolysate were assessed. Yield of protein hydrolysate was ~13.5 % of wet weight of skin and has the molecular weight <6kDa. The yield of keratin hydrolysate was ~3.5% with a molecular weight <500 Da.

**Example 5**

One kg of wet salted raw trimmings of bovine origin were cut into pieces and washed four times with 2 lit of water to remove dirt and salt for 4 hrs. The washed trimmings were loaded into reactor and treated with 2 lit of 3 % (v/v) of acetic acid at 60 °C. After a period of 5 hrs, 9 lit of 3 % (v/v) of acetic acid was added continuously to the reactor at constant flow rate of 10 mL/min and the reaction displaced gelatin solution from the reactor for period of 18 hrs with stirring at 800 r.p.m. The temperature of the extraction was maintained at 60 °C. The obtained gelatin solution was pre-filtered using 5 micron cartridge and purified by ultra filtration using ceramic filter of size 0.8 micron, concentrated with nano filter of size 400 Da and dried. The left over skin with hair matrix were enzymatically hydrolysed using 18 g of acid protease enzyme at ~38°C for 6 hrs to obtain protein hydrolysate. This residual hair added with 0.75 lit of water and treated with 10 g of alkali NaOH at 120°C with 15 psi for 2 hrs to obtain keratin hydrolysate. Yield of extracted gelatin was determined using hydroxyproline estimation and the physicochemical properties such as gel strength, viscosity were characterized. Similarly, the yield and properties of protein, keratin hydrolysates were determined. Here, the yields of the three products were estimated on the basis of wet weight of the raw trimmings. Yield of the gelatin was ~18% and other properties such as gel strength and viscosity are ~225 blooms and 3.4 cP respectively. Similarly, yield and physicochemical properties of the protein hydrolysate and keratin hydrolysate was assessed. Yield of protein hydrolysate was ~12.8% of wet weight of trimmings and has the molecular weight <6kDa. The yield of keratin hydrolysate was ~ 4.8% with a molecular weight <340 Da.

**Example 6**

One kg of wet salted raw trimmings of bovine origin were cut into pieces and washed thrice with 3 lit of water to remove dirt and salt for 4 hrs. The washed trimmings are loaded into reactor and treated with 2.5 lit of 4.5 % (v/v) of acetic acid at 65 °C. After a period of 5 hrs,
9 lit of 4.5 % (v/v) acetic acid was added continuously to the reactor at a constant flow rate of 10 mL/min for 15 hrs and 3.5% of fat removal was achieved. The obtained gelatin solution was pre-filtered with 5 micron and purified by ultra-filtration using ceramic filter of size 0.8 micron. Then, it was concentrated with nano filter of size 400 Da and then dried. The left over solution with hair and skin matrix was enzymatically hydrolysed using 15 g of acid protease at 38°C for 4 hrs and filtered to obtain protein hydrolysate. Residual hair present in the reactor was added with 1 lit of water and treated with 6 g of KOH at 120°C for 2.5 hours with the pressure of 15 psi. Yield of extracted gelatin was determined using hydroxyproline estimation and the physicochemical properties such as Gel strength, Viscosity were characterized. Yield of the gelatin was 20% of wet weight of skin with gel strength of ~280 blooms and viscosity of 8.0 cP. Similarly, yield and physicochemical properties of the protein hydrolysate and keratin hydrolysate were assessed. Yield of protein hydrolysate was ~12.3 % and as the molecular weight <6kDa. The yield of keratin hydrolysate was ~4.8% with a molecular weight <450 Da.

Example 7

Example 8
into reactor and heated to 65 degree C in the presence of water for 1 hour. After a period of 1 hr, the temperature is brought to 55 degree C and enzymatically hydrolysed using 40 g of alkali protease enzyme for 6 hrs to obtain Collagen hydrolysate. The residual matrix with hair is added with 0.75 lit of water and treated with 10 g of alkali NaOH at 120°C with 15 psi for 2 hrs to obtain keratin hydrolysate. Here, the yields of the collagen hydrolysate and keratin hydrolysate were estimated on the basis of wet weight of the raw trimmings. Yield of collagen hydrolysate was ~25% and with the molecular weight <6kDa. The yield of keratin hydrolysate was ~ 5% with a molecular weight <600 Da.

ADVANTAGES OF THE INVENTION

➢ Provides a process for complete utilization of raw hide/skin trimming wastes without any series of time consuming pretreatment, which is more than a week for conventional process. In the present invention, the extraction of products could be completed in simple steps within 2 days.

➢ The process provides three important products, namely gelatin, protein hydrolysate containing both collagen as well as keratin, and keratin hydrolysate

➢ Provides a simple and more effective method for making three products such as gelatin, protein (collagen) hydrolysate and kertain hydrolysate using thermo-chemical, enzymatic and thermo-chemical hydrolysis respectively, through a continuous mode of extraction within 48 hrs.

➢ Provides an option for extraction of gelatin prior to the removal of hair by liming process.

➢ Process provides a continuous mode of extraction for gelatin, which will lead to hydrolysis of skin collagen in controlled manner to give pharmaceutical/food grade gelatine

➢ Process is a simple method, which excludes all time consuming processes used conventionally.

➢ Provides an environment friendly and economically viable process.
Process finds potential application in the manufacturing of pharmaceutical/food grade gelatin, protein (collagen) hydrolysates which can be used for pharmaceutical/food applications or fertilizer for agricultural applications and keratin hydrolysate can be used as filler for leather manufacture or other applications such as fertilizer, cosmetics and pharmaceutical formulations.
**CLAIMS:**

1. A process for the treatment of raw hide/skin trimmings, wherein the steps comprising:

   [i] treating raw hide/skin trimming with 5 to 90% (w/w) of an organic acid at a temperature in the range of 45 to 70 degree C for a period in the range of 15 to 24 hours followed by separation of the resulting solution and subsequent drying at a temperature in the range of 4 to 70 degree C to obtain gelatin;

   [ii] hydrolysing the residual skin matrix as obtained in step [i] or raw hide/skin pretreated at 60-80 degree C; with 0.1 to 10% (w/w) of an enzyme at pH ranging from 2.5 to 10 in the presence of 100 to 300% (w/v) of water at a temperature ranging from 35 to 60 degree C for a period in the range of 4 to 10 hours followed by treatment, as an optional step, with 2 to 15% (w/w) of caustic alkali in the presence of 100 to 300% of water at 1 to 5 atmospheric pressure and separating the solution from the matrix and drying at a temperature in the range of 45 to 150 degree C to obtain protein hydrolysate containing both collagen as well as keratin;

   [iii] treating the residual hair obtained after separation in step [ii] with 2 to 15 % (w/v) of a caustic alkali in the presence of 50 to 150% of water at a temperature ranging from 90 to 120 degree C for a period in the range of 3 to 12 hours under 1 to 5 atmospheric pressure followed by separation of the resulting solution and subsequent drying at a temperature in the range of 45 to 150 degree C to obtain keratin hydrolysate.

2. The process as claimed in claim 1, wherein the organic acid used in step (i) is selected from the group consisting of acetic acid and propionic acid.

3. The process as claimed in claim 1, wherein the enzyme used in step (ii) is selected from the group consisting of acid protease, neutral protease and alkali protease.

4. The process as claimed in claim 1, wherein the method of separation is selected from filtration and centrifugation.

5. The process as claimed in claim 1, wherein the pore size for separation is in the range of 0.8 to 2 microns.
Sequential stages for extraction of high valuable products from trimmings

Stage 1
Continuous mode of gelatin extraction by acid/thermal hydrolysis

Stage 2
Process leave residual skin matrix with hair

Stage 3
Process leave residual hair

Residual skin matrix/Processed raw hide/skin treated with protease enzyme

Residual hair treated with alkali (e.g., NaOH, KOH)

Product obtained in individual stage, dried and characterized

Used in various field like pharma, food, cosmetic, leather, animal feed industries

Fig. 1

Raw materials washed with water

Hide and skin trimming from tannery/daughter house

Existing process

Extraction of valuable products in individual form

Removal of fat

Raw material pretreated (48hrs-3 weeks) with various chemical agents (e.g., lime, Na₂S, HCl, CaCl₂, and H₂O₂)

Removal of hair

Extraction of gelatin from pretreated skin/hide

Extraction of protein hydrolysate

Purification and characterization of the protein hydrolysate

Used as animal feed, leather auxiliary

Batch extraction of gelatin by acid/thermal hydrolysis

Gelatin purification and characterization

Used in pharma, food, cosmetic industries

Pretreated hair was hydrolysed with 20% alkali for 2-20 hrs

Fermentation and characterization of keratin hydrolysate

Used as leather filler, synlan