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3,738,913

**METHOD OF PREPARING POLYPEPTIDES**

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 No Drawing. Continuation-in-part of application Ser. No. 548,374, May 9, 1966, which is a continuation-in-part of application Ser. No. 301,970, Aug. 14, 1963, now abandoned. This application Sept. 11, 1970, Ser. No. 71,355

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7 Claims

**ABSTRACT OF THE DISCLOSURE**

The method of producing the polypeptides of this invention deals with protein hydrolysis wherein a collagen-containing material is subjected to heat in the presence of aqueous solution having sulfite ions present, to produce a hydrolyzed protein solution of reduced gel-forming character having a Formol Nitrogen Value of the order of 6 or less, subjecting the hydrolyzed protein solution to anion exchange treatment and thereafter further hydrolyzing the proteins using proteolytic enzymes to produce a polypeptide product having a Formol Nitrogen Value in the range of 8 to 17, which polypeptides are useful for treatment of hair due to unique sorption characteristics.

This application is a continuation-in-part of application Ser. No. 548,374, filed May 9, 1966, and entitled Proteinaceous Cosmetic Material, now abandoned, which application was in turn a continuation-in-part of application Serial No. 301,970 entitled Proteinaceous Food Material and Method of Preparing Same, filed Aug. 14, 1963, and now abandoned.

This invention relates to a method of preparing polypeptide products from collagen-containing material. More particularly, it relates to a method of producing non-antigenic proteinaceous materials having utility in cosmetics such as for the treatment of skin and hair, with characteristics rendering the products uniquely suitable for use in the care of hair, i.e., hair grooming and cleansing compositions such as setting lotions, tonics, bleaches, shampoos, and the like.

In accordance with this invention, a method of converting the proteinaceous components of collagen-containing materials to non-antigenic polypeptides having unique absorbability characteristics rendering the polypeptides useful in cosmetics, is provided which involves preparation of hydrolyzed protein of a Formol Nitrogen Value generally of the order of 4 to 7 through subjecting collagen-containing material to heat in the presence of aqueous solution having sulfite ions present therein under conditions of temperature, pressure and time whereby the gel-forming character of the hydrolyzed protein is at least appreciably reduced, the hydrolyzed protein in solution form is contacted with anion exchange material whereby coloring elements or color precursors are removed and then further hydrolyzing the proteins of reduced gel-forming character with enzymes having proteolytic activity at temperatures in the range between 100° F. and 180° F. to produce polypeptides of Formol Nitrogen Value in the range between about 8 and 17 and heat treating the product of enzyme hydrolysis at temperatures in the range between about 195° F. and 215° F. to arrest the enzyme action.

**DISCUSSION OF THE PRIOR ART**

In many uses of proteinaceous materials, for example, in cosmetic and pharmaceutical products, the protein additive must be low in color, bland in odor, and low in ash.

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These requirements can be met by most collagen derivatives such as gelatin. Use of gelatin in, for example, cosmetics, however, generally produces undesirable texture due to high viscosity and stable gel-forming ability.

Modification of the properties of gelatin by hydrolysis has been suggested in Lewis Pat. No. 3,016,334 and Keil et al. Pat. No. 2,431,256. Lewis suggests producing a product useful in skin creams by hot water hydrolysis of proteins from porkskins to obtain material of intermediate ratings, i.e., gel strength (bloom zones) 40 to 80 and millipoise viscosity of 12 to 20 which products have a Formol Nitrogen Value of less than 5.

Keil et al. Pat. No. 2,431,256 teaches hydrolysis of proteins with acids, alkalis and enzymes followed by pH adjustment of the hydrolyzate to precipitate the higher molecular weight fractions to produce foam stabilizers. Digestion of proteins using the suggested enzyme treatment, i.e., 1% to 4% of papain for from 3 to 24 hours reduces the proteins to amino acids and to polypeptides of molecular weight well below 500, i.e., Formol Nitrogen Value well above 17.

**THE PRESENT INVENTION**

Now we have discovered that polypeptide material which when used in cosmetics, for example, hair grooming compositions, is capable of sorption into the hair, imparts gloss, renders the hair manageable and acts as a humectant or moisturizer, can be produced by processing collagen to obtain a proteinaceous extract whose components are of a specific molecular weight size range. This processing, for example, may convert collagen directly to a non-gelling material by the one step process of subjecting raw material which contains collagen to high temperatures and pressures in an aqueous system or indirectly by first preparing gelatin and then destroying its gelling character by subjecting it to heat and water, and thereafter the proteinaceous material is hydrolyzed with proteolytic enzymes to the degree required to have a product having a Formol Nitrogen Value in the range between about 8 and 17, characterized by being straight chain polypeptides free of sulfur-containing amino acids and completely soluble in aqueous media over the entire pH range of 1 to 12.

In a preferred embodiment of the invention, the collagen-containing material and associated proteins found in such sources as bones, skins, hides, sinews, fatty tissues, and in specific materials, such as pigs' feet or ossein which are preferred sources of proteins because the end products are clear, more stable solutions having a better odor, is treated in aqueous solution with a sulfite agent to minimize development of color during heating and solubilization and the hydrolyzed protein obtained through treatment with heat and water having a Formol Nitrogen Value of less than 8 and then is subjected to the hydrolysis action of the proteolytic enzyme, papain, to reduce the polypeptides to a mixture of proteinaceous materials having a Formol Nitrogen Value in the specified range, preferably in the range between 9 and 14. In those instances where a light color and bland odor are highly desirable such as in hair grooming compositions, the components present in the hydrolyzed material which cause the solid proteinaceous products to darken during drying and which are responsible for off odors, can be largely removed by solvent extraction with alcohols, by following hydrolysis of the collagen-containing material in the presence of sulfur dioxide with a peroxide treatment for alteration of sulfur compounds or by contacting the product of hydrolysis in the presence of sulfur dioxide with ion exchange materials, either prior to or subsequent to the enzyme hydrolysis, etc.