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WAVESET COMPOSITION COMPRISING AN AQUEOUS LOTION OF POLYVINYL ALCOHOL AND A CARBOXY VINYL POLYMER

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This invention relates to new compositions of matter and to methods for their employment in hair-dressing. More particularly, it relates to waveset lotions by the use of which human hair may be caused to retain for an extended period of time a desired configuration imposed on it by means of curlers, pins, combs, nets, or other apparatus.

A very large number of hair-waving compositions have been proposed. In terms of their mode of action and application, these may be classified into three broad classes. One class includes those which operate chemically by breaking disulfide (—S—S—) linkages in the hair protein, thus temporarily relaxing the hair so it can be shaped or set as desired, and then establishing new disulfide linkages to restore most of the original strength to the hair in the artificially imposed configuration. A second class includes those which act largely by swelling and softening the hair so that, after curling while swelled and softened and then dried under tension, it retains a curliness due to the partial flattening of the normally circular cross-section of the individual hairs. The third class includes those preparations which leave a thin layer of a film-forming material on the individual hair filaments; when the thus coated hair is dried in a desired configuration imposed by curlers, pins, combs, nets, etc., the microscopically thin layer of film-forming material tends to retain the shape in which it dried and thus resists the tendency of the hair to resume its initial configuration. A recently introduced sub-group within this third class comprises the aerosol lacquers which employ quick-evaporating solvent solutions of resins such as shellac and polyvinylpyrrolidone.

A certain amount of overlapping may exist as among these three classes; i.e., a hair wave preparation may combine, to a greater or lesser extent, two or more of the above modes of action. The compositions of the present invention belong in the second and third categories discussed above; they soften and swell the hair filaments to a certain extent, and leave a microscopically thin resinous film on the individual hair which dried film tends to stabilize the configuration of the hair.

In the past, waveset lotions of this class have ordinarily been composed essentially of water solutions of natural gums such as quince-seed, karaya gums, and the like. Employment of these natural gums has resulted in several serious disadvantages. Being of natural origin, these gums tend to vary a great deal from batch to batch, so that manufacture of a lotion of uniform properties has been difficult. Furthermore, these natural gums support the growth of various micro-organisms and are difficult to preserve; even with preservatives present, they tend to suffer gradual chemical deterioration and decomposition, so that the shelf life of waveset lotions containing them may be limited to about three to six months.

To overcome these drawbacks to the use of natural gums, the use of certain synthetic film-forming gums has been proposed. Among these are polyvinylpyrrolidone and various cellulose ethers, such as methyl cellulose. Unfortunately, when used in quantities to provide an adequately stiff film, these tend to be objectionably sticky (polyvinylpyrrolidone) or to leave flaky or linty particles on the hair.

We have found that excellent waveset lotions, which have the surprising advantage of eliminating flakiness in use, may be prepared by combining water with (1) from about 0.1 to about 2.5 percent of its weight of polyvinyl alcohol, (2) from about 0.02 to about 2.5 percent of its weight of a particular type of very slightly cross-linked carboxyvinyl polymer of extremely high molecular weight and having an acid equivalent weight of about 73 to 76 and capable, when at least partially neutralized, of greatly increasing the viscosity of aqueous systems, and (3) enough of a base to bring the pH into the range 4.5 to 8.5, and preferably into the range 6 to 7.5. Such a carboxyvinyl polymer is available from the B. F. Goodrich Chemical Company under the trademark designation "Carbopol 934," and has been described as a water-soluble polymer of acrylic acid cross-linked with 1% of a polyallyl ether of sucrose having an average of about 5.8 allyl groups for each molecule of sucrose. Similarly constituted products designated as Carbopol 940 and Carbopol 941 may also be employed; the former is similar in most properties to Carbopol 934 but gives solutions of somewhat improved clarity; Carbopol 941 gives slightly less viscous solutions than Carbopol 934 and Carbopol 940, but is entirely suitable for use in this invention.

When set with these waveset lotions, the hair stays in place well during the drying process. On combing out, the hair combs easily, with no flaking, and the waves fall into place nicely. The waves stay in the hair as long or longer than when set with waveset lotions made with natural gums. An important advantage our new waveset lotions possess is their great storage stability. They are not subject to microbial attack, and retain their viscosities substantially unchanged for indefinite storage periods.

While no preservative is needed to preserve our waveset lotions from the time of manufacture up to the time of use, we usually add a small amount of preservative to prevent microbial growth which could be nourished by nutrients introduced into the lotion by dipping of combs, brushes or the like into the lotion.

A further advantage of our novel waveset lotion is the entire absence of the rather repulsive slimy feel which has characterized many prior art waveset lotions.

While we do not understand fully the mechanism of the remarkable hair-stabilizing properties of our novel wave-set lotions, we believe that the most important ingredient in the dry film is the polyvinyl alcohol, and that it is chiefly this which gives the desirable maintenance of configuration of the set hair. The crosslinked carboxyvinyl polymer (e.g., a Carbopol) increases the viscosity of the lotion, and thus, we believe impedes premature draining of the lotion from the hair and so aids in the formation of a film of effective thickness.

Polyvinyl alcohol, as is well known, is prepared by polymerizing vinyl acetate to a very high molecular weight polymer and then hydrolyzing off at least a major proportion of the acetate radicals. While completely hydrolyzed grades of polyvinyl alcohol may be used, we prefer to employ those which are only about 80–95 percent hydrolyzed, since these exhibit better cold-water solubility and have less tendency to form semi-solid gels in water on prolonged standing. We also prefer to employ polyvinyl alcohols of intermediate viscosity. We have found that "Elvanol 52–22" polyvinyl alcohol, supplied by E. I. du Pont de Nemours and Co., Inc., to be especially useful in our invention. This material has a viscosity of 21 to 25 centipoises at 20° C. in a 4 percent water solution, is 87–89 percent hydrolyzed, has a pH of 6–8, and contains no more than 5 percent volatiles and no more than 0.75 percent ash as Na_2O. Polyvinyl alcohols having viscosities as low as about 5 and as high as about 60 centipoises at 20° C. in 4 percent solutions are also suitable, though less preferred.

It is essential that the high molecular weight carboxyvinyl polymer be at least partially neutralized with a base; this partial neutralization should bring the pH into the range of about 4.5 to 8.5, preferably about 6–7.5, and greatly increases the viscosity of the system. The choice of a base for this partial neutralization of the high molecular weight carboxyvinyl polymer (e.g., "Carbopol 934") is not critical, and any water soluble base whose ions are non-toxic may be used; sodium hydroxide, potassium hydroxide, ammonia, triethylamine, di-isopropanolamine, tri-isopropanolamine, diethanolamine, triethanolamine, N-hydroxyethylmorpholine, etc., are suitable. We prefer to use triethanolamine or sodium hydroxide.

As above pointed out, we normally include a small amount of preservative to prevent growth of bacteria or molds on debris which might be introduced into the lotion in course of its use, as by dipping a brush or comb into it. We prefer to use butyl p-hydroxybenzoate, though other preservatives well known in the art may be used instead. For example, methyl p-hydroxybenzoate or propyl p-hydroxybenzoate may be used.

Optionally, small amounts of perfume and/or color may be incorporated in our waveset lotion if desired.

The following compositions illustrate waveset lotions of our invention. All parts are by weight.

Examples 1 and 2

	Example 1	Example 2
Polyvinyl Alcohol (Elvanol, Type 52–22)	0.500	0.500
Carbopol 934	0.180	0.160
Butyl p-hydroxybenzoate	0.018	0.0180
Triethanolamine	0.2160	0.1920
Perfume	0.0529	0.0529
Color Solution	0.1746	0.1746
Water	98.8585	98.9025
pH	7.0	7.0
Viscosity, (No. G–4 Zahn Cup)—seconds	20	16

Waveset lotions of Examples 1 and 2 may be used at their full strength or may be diluted with as much as four or five parts of water. A more dilute preparation than those of Examples 1 and 2, which is suitable for easily set hair is that of the following Example 3.

Example 3

Polyvinyl alcohol (Elvanol, Type 52–22)	0.100
Carbopol 934	0.030
Butyl p-hydroxybenzoate	0.018
Triethanolamine	0.036
Water	99.816

The following table shows other wave-set compositions we have made in accordance with this invention.

Examples 4–11

Example No	4	5	6	7	8	9	10	11
Carbopol 934	0.16	0.16	0.18			0.16	0.16	0.16
Carbopol 940				0.18				
Carbopol 941					0.18			
Triethanolamine	0.192			0.216	0.216	0.192	0.192	0.192
Sodium hydroxide		0.068	0.0765					
Polyvinyl Alcohol:								
Elvanol, grade 52–22	0.35	0.35	0.35	0.50	0.50			
Elvanol, grade 51–05						0.35		
Elvanol, grade 72–60							0.35	
Elvanol, grade 50–42								0.35
Perfume	0.053	0.053	0.053	0.053	0.053	0.053	0.053	0.053
Color solution	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17
Butyl p-hydroxybenzoate	0.018	0.018	0.018	0.018	0.018	0.018	0.018	0.018
Water	99.05	99.18	99.15	98.86	98.86	99.06	99.06	99.06
pH	7.09	7.62	7.49	7.49	6.79	6.28	6.28	7.12
Viscosity (G–4 Zahn Cup), seconds	14	13	16	20	12	9.5	11	10.5

In the above table the polyvinyl alcohols have properties as summarized in the following table:

	Viscosity,[1] cp.	Percent Hydrolysis	pH	Volatiles, percent Maximum	Ash, percent Max. (as Na_2O)
Elvanol 52–22	21–25	87–89	6–8	5	0.75
Elvanol 51–05	4–6	88–89	6–8	5	0.75
Elvanol 72–60	55–65	99–100	6–8	5	1.00
Elvanol 50–42	35–45	87–89	6–8	5	0.75

[1] Viscosity of a 4% water solution at 20° C. determined by the Hoeppler falling ball method.

We claim:

1. A waveset lotion comprising water, polyvinyl alcohol, and a high molecular weight carboxyvinyl polymer comprising polyacrylic acid crosslinked with about 1% of polyallyl sucrose having an average of about 5.8 allyl groups for each sucrose molecule, said carboxyvinyl polymer being partially neutralized by a water soluble base, the said polyvinyl alcohol being present in an amount of between 0.1 and 2.5 percent, the said carboxyvinyl polymer being present in an amount between 0.02 and 2.5 percent.

2. A waveset lotion according to claim 1 in which the carboxyvinyl polymer has been partially neutralized with triethanolamine, said lotion having a pH of between about 4.5 and about 8.5.

3. A waveset lotion according to claim 1 in which the carboxyvinyl polymer has been partially neutralized with sodium hydroxide, said lotion having a pH of between about 4.5 and about 8.5.

References Cited in the file of this patent

Ruemele: Manufacturing Chemist, January 1952, p. 25.

Carbopol 934, 11 p. booklet of B. F. Goodrich Chem. Co., Cleveland, Ohio, Service Bulletin G.C.–20, March 1954, pp. 4 and 11.

Carbopol 934, 24 p. booklet of B. F. Goodrich Chem. Co., Cleveland, Ohio (December 27, 1957, pp. 6 and 10).