The development of hair styling products with new " Aspartate polymer (Poly amino acid derivative) "

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SYNOPSIS

There have been many kinds of hair styling sprays with various setting effects. Consumers have used strong setting effect hair sprays to get a long lasting hold. In recent years, however, more and more consumers have come to prefer a "soft & natural" touch feeling, keeping the same long lasting hold. Nonetheless, the existing approaches to this feature could not respond to the consumers' needs, since products lose the hold strength if the soft feeling is pursued, and vice versa. We have researched and developed a new products to attain a compatible feature with both long lasting hold and natural feeling.

Then, we have developed a new multifunctional hair styling material "Poly Amino Acid Derivative (PAAD)." We have focused on the PAAD's feature that highly diffuses onto a hair and makes thin and even layer on a hair, and have made trials and errors to improve holding strength. "PAAD" excellently makes hair memorize its curl shape which is as the same effect as existing ordinary acrylic resin. Further more, it leaves a soft and natural touch feeling on the hair. We have accomplished a new Poly Amino Acid Derivative with ambivalent features, "soft & natural finish" and "long lasting hold ", and now we report about it.

INTRODUCTION

Consumers have been looking for strong setting effect hair styling sprays for a long time. In recent years, however, consumers have come to prefer a "soft & natural " touch feeling .The objective of the work described here was to address this consumer concern and to develop a novel hair styling product capable of attaining a compatible feature with both long lasting hold and natural feeling. We added various plasticizers to existing ordinary acrylic resin, first.

However in this approach, we could not achieve our aim. In previous work carried out in our laboratory, it was determined that the formation of thick and uneven film on the hair caused the feeling of stiffness, besides the fact that polymer film had a hard texture. On the basis of this hypothesis, we set about to make a thin, homogeneous, and strong film on the hair, which would be the most effective way to give a natural touch feeling as a finish, keeping a long lasting hold.

Therefore we set our goal to develop a new ingredient with high diffusing ability on the hair for making the thinner film. To deal with this challenging issue, we started with designing of polymers which was able to form a thin, homogeneous and strong film on the surface of individual hair fiber.

Poly amino acid is known for its high diffusing ability on the hair. (Amino acid is a component of the hair) Taking advantage of this property we thought that it would be possible to attain a compatible features with both long lasting hold and natural feeling. This new approach to cosmetics is the heart of this study.

We have studied the correlation between the structure of polymers and their effects on the hair by synthesizing various polymers incorporating the monomer of amino acid derivative, and evaluating the properties of treated hair.

This study allowed us to determine the optimal polymer structure .

EXPERIMENTAL

(1) Measurement of diffusing ability

The diffusing ability was confirmed as followed: First, 5% polymer solution in ethanol (1g) was dropped on the wool muslin which has similar surface characteristic with human hair. After 5 minutes, the diameter of the droplet was measured.

In order to confirm the forming condition of the film on the actual hair, we also observed a hair sample immersed in 5% polymer solution in ethanol for 1sec, followed by drying at 55℃ for 40min. by electron microscope (×200, S-4100, HITACHI, LTD, Japan).

(2) Measurement of film strength

We defined the strength of a film as breaking strength under physical tension. The strength of the film was measured by rheometer. (CR-200D, SUN SCIENTHIC CO.LTD, Japan) The film was formed by 5% polymer solution in ethanol drying at 60 ℃ for 60min.Then, thickness of the film was adjusted to be 0.5 mm.

(3) The feeling of stiffness of the treated hair

The stiffness of the hair against bending force was measured by rheometer (CR-200D, SUN SCIENTHIC CO.LTD, Japan) using a hair tress. The instrument is depicted in Fig.1. The treatment procedure of hair tress is as follows:

Tresses (2g, 10cm) were treated with 5% polymer solution in ethanol (0.5g). After tresses were left at 55 ℃ for 60 min, durability against vertical power is measured by rehometer .

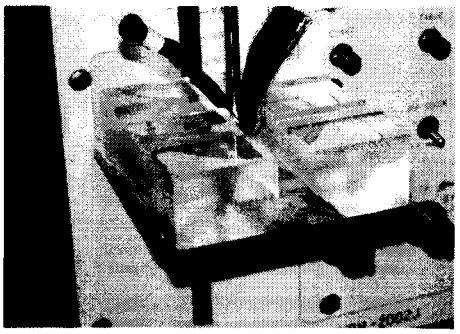


Fig.1 Measurement of the feeling of stiffness

(4) Curl retention test

Tress (2g, 18cm) was rolled up around Teflon stick, and treated with 5% polymer solution in ethanol (0.5g), followed by drying at 55 ℃ for 60min.

The setting effectiveness of polymers at high humidity (for 3h, 25 ℃, RH 95%) and under some physical tension (for 10min, shaking by 120times/min.) were measured by curl retention.

(5) Measurement of moisturizing effect

Each terminal of 30 hair fibers was parallely fixed on water repellent papers at equal distances without overlap. The distance between two papers (exposed length of hair fibers) was 10cm. The hair sample was immersed in 5% polymer solution in ethanol for 1sec, followed by drying at 25℃, RH 65% for 120min. After drying, the hair sample was set under fixed humidity condition (RH 35%, RH 65%, RH95%) for 5h. The remaining water content was measured by Karl-Fischer Moisture Titrator (MKA-210 KYOTO ELECTRONICS. INC, Japan). Using the Karl-Fischer method, we measured the remaining water content of the hair itself (A), by subtracting remaining water content of the just polymer (B) from that of the whole polymer- treated-hair (C) under fixed humidity conditions.

$$A = C - B$$

In addition, the change in calorie was measured by Differential Scanning Calorimeter (DSC220C SEIKO INSTRUMENTS. INC, Japan.) for thermal analysis.

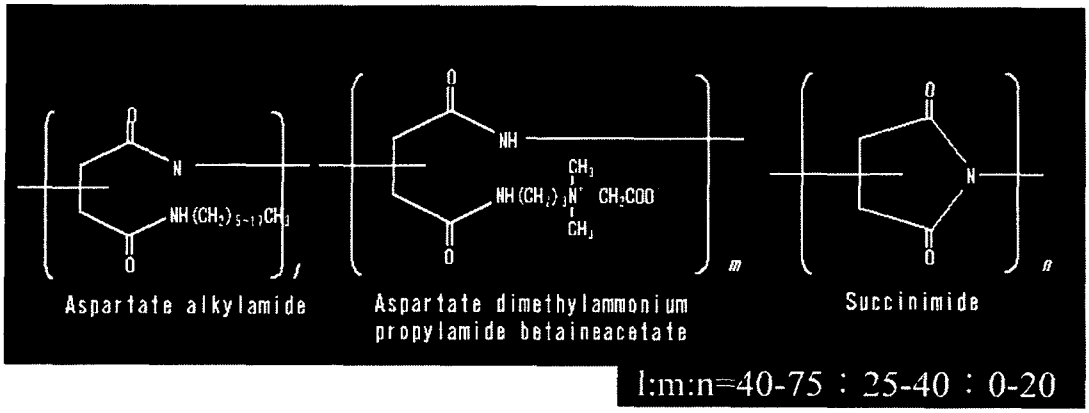
RESULT AND DISCUSSION

Polymer design

First, we prepared the initial Aspartate polymer sample in order to understand the capability of Poly amino acid derivative, to clarify whether the polymer forms thin and homogeneous film. The result confirmed that the initial sample formed a thin and homogeneous film, and it didn't have stiffness in feeling. However, there were significant points to be improved. The strength of the film itself and the durability against wetness (high humidity conditions) were weak. We performed polymer design in order to improve durability against high humidity and strength of the film. By evaluating the influence of "molecular weight", "the incorporating of alkylamide group", and "the incorporating of betaine group", we have studied improving durability against high humidity and strength of the film. Thus, we have succeeded in developing a novel polymer "Poly amino acid derivative (PAAD)"

Shown below (Fig. 2) is a structure of "PAAD". "PAAD" consist of Aspartate alkylamide, as a main monomer, Aspartate dimethylammonium propylamide betaineacetate group, Succinimde group. And it is a polymer with a molecular weight of about 200,000.

Fig.2 The structure of "PAAD"



Performance as a novel polymer "PAAD" itself

We have confirmed if the finalized "PAAD" itself has met expected function. We have used wool muslin which has a similar surface characteristics to human hair, and have confirmed diffusing ability by measuring the diameter after dropping the polymer solution. Fig.3 shows the degree of diffusing ability. It was confirmed that "PAAD" had higher diffusing ability compared to existing acrylic polymer.

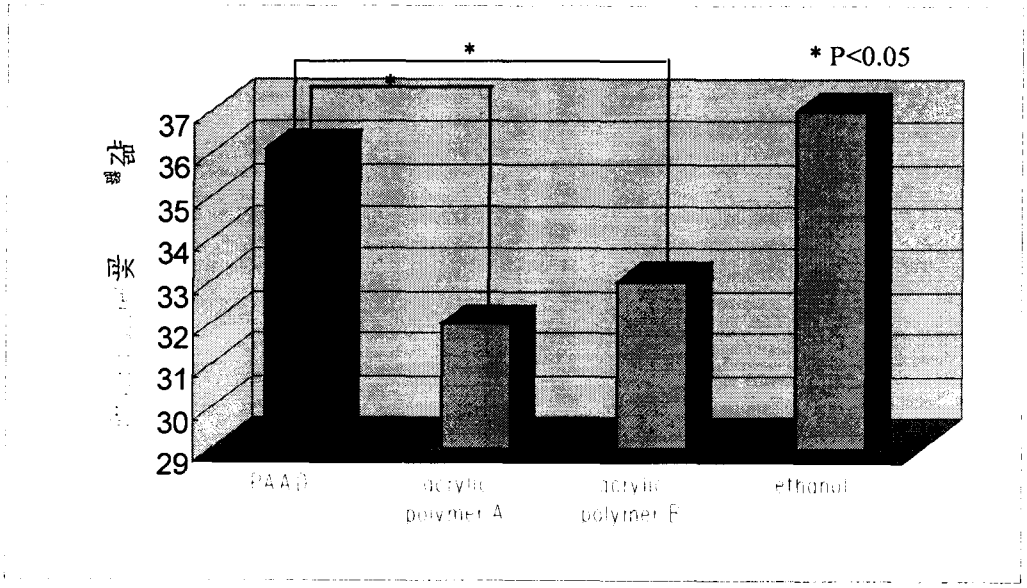


Fig.3 The diffusing ability of the polymer. 5% polymer solution in ethanol (1g) was dropped on wool muslin. After 5 min, the diameter of the droplet was measured

We also observed the condition of human hair treated with the polymer solution by electron microscope (\200). In comparison to untreated hair, the hair treated with existing acrylic polymer was thickly coated, and we couldn't see the cuticle (fig.4-B). On the other hand, since "PAAD" formed a thin and homogeneous film, we could see the cuticle through the "PAAD" film (fig.4-C).

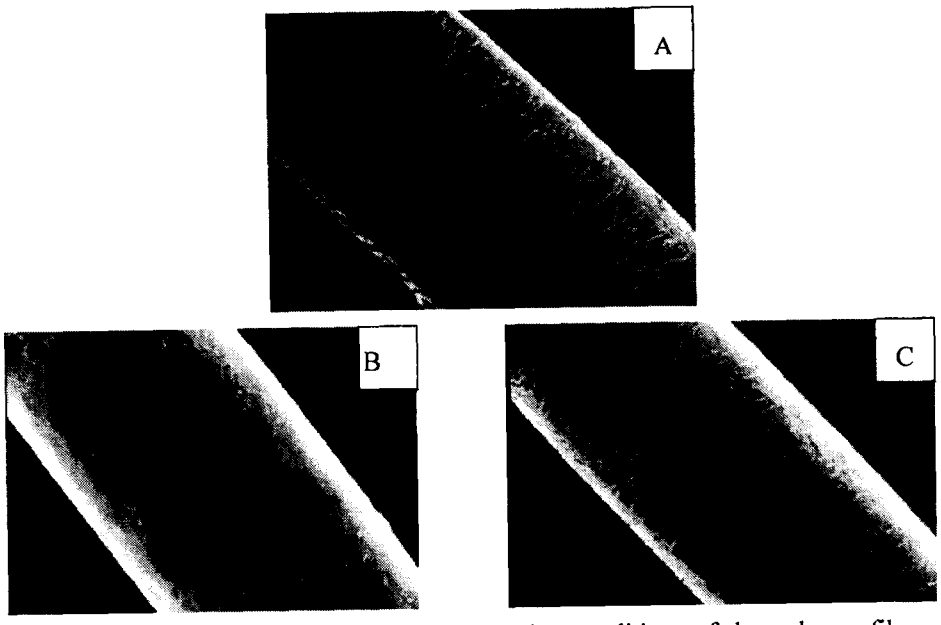


Fig.4 Electron microscopic observation. The forming conditions of the polymer film on the hair were observed by electron microscope. (×200, S-4100, HITACHI, LTD, Japan) The hair was immersed for 1 sec, in polymer solution.
A: untreated hair, B: treated by existing acrylic polymer, C: treated by "PAAD"

Next, the strength of the polymer film was measured by rehometer. Fig.5 shows the breaking point (g) per certain film volume. It was confirmed that PAAD's film had higher durability against stress (weight), compared to existing acrylic polymer.

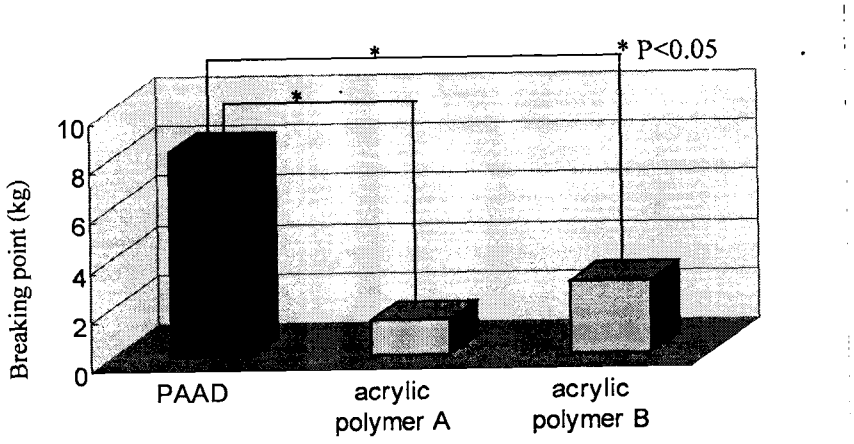


Fig.5 The strength of the polymer film. We defined it as breaking point under physical tension, was measured by rheometer (CR-200D, SUN SCIENTHIC CO.LTD, Japan), using the hair tress. The thickness of the polymer film was adjusted to be 0.5mm.

Performance of the hair treated by novel polymer "PAAD"

From the above evaluation, it was confirmed that this novel polymer "PAAD" met the expected function. Furthermore we evaluated the stiffness and hold strength of the hair treated by the polymer. The stiffness of the hair was measured by rehometer, using hair

tresses. We found out that the feeling of stiffness and the stress value of the tress were correlated. The tress treated by "PAAD" had a lower bending point (g) and less feeling of stiffness compared to the tress treated by existing acrylic polymer. (Fig.6)

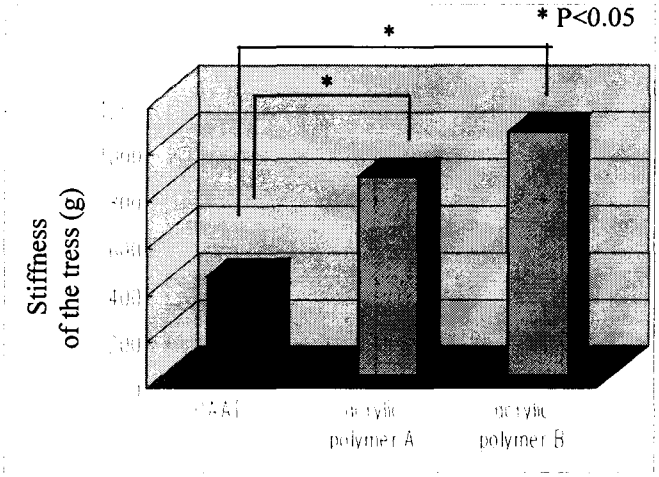


Fig.6 The stiffness of the hair against a bending force. It is confirmed that "The feeling of stiffness on the hair" has a mutual relationship to durability against vertical force.

Generally, styling products, which don't have the stiffness in feeling, tend to have weaker setting effect. We used the curl retention method which is ordinary used to evaluate the setting effect. By this method, we have evaluated setting effectiveness against high humidity and physical tension. The setting effect of the hair treated by "PAAD" showed similar durability against physical tension and higher durability against humidity in comparison to the hair treated by existing acrylic polymer (Fig.7, 8). In comparison to existing acrylic polymers, "PAAD" can form thinner, more homogeneous, and stronger film. Thus, it can attain compatible features with both long lasting hold and natural feeling.

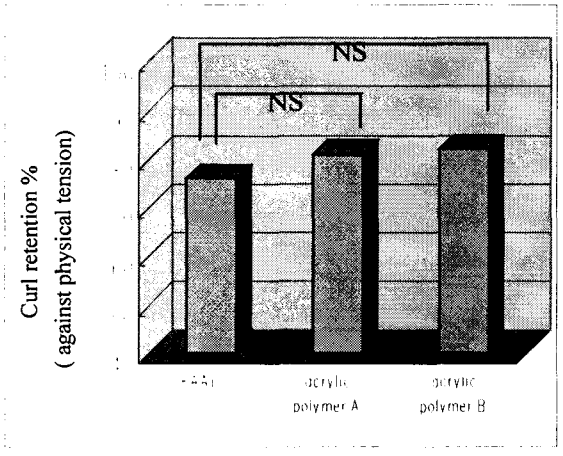


Fig.7 The setting effectiveness under physical tension.

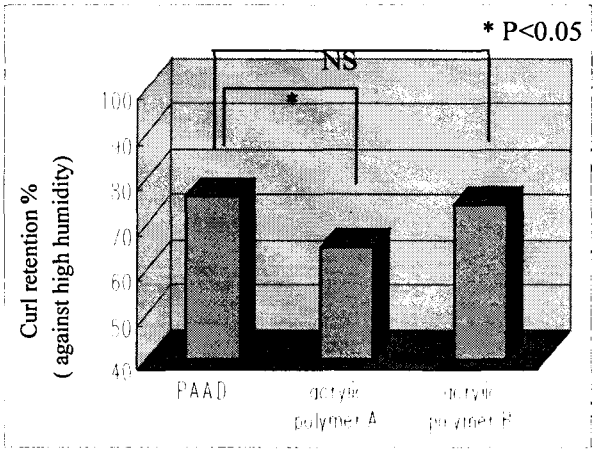


Fig.8 The setting effectiveness under high humidity condition.

Other benefits of "PAAD"

Besides performing as film-forming polymer, "PAAD" , as an active ingredient, has other benefits. As previously shown in the structure formula, The main chain of "PAAD" consists of Aspartate , mainly, is considered to have moisturizing effect, same as Poly amino acid which has been applied as moisturizer. Therefore, we evaluated moisturizing effect using Karl-Fischer Moisture Titrator. Fig.9 shows the retaining water content of the hair itself. It was determined that "PAAD"-treated hair contained higher remaining water content under the drying conditions, compared to existing acrylic polymers. It was also confirmed that it contained close to the same remaining water content as existing moisturizer of polymer type.

In addition, we used Differential Scanning Calorimeter to measure the change in calorie of the polymer-treated-hair which were left under several humidity condition. We assume that the change in remaining water content (the trans epidermal water loss) corresponds to change in calorie. Considering the result of the remaining water content and thermal analysis, "PAAD" reduced "the water loss" from the hair under drying conditions compared to existing acrylic polymers. (Fig. 9,10)

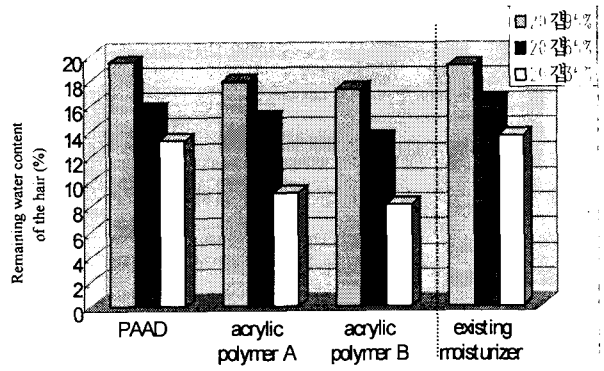
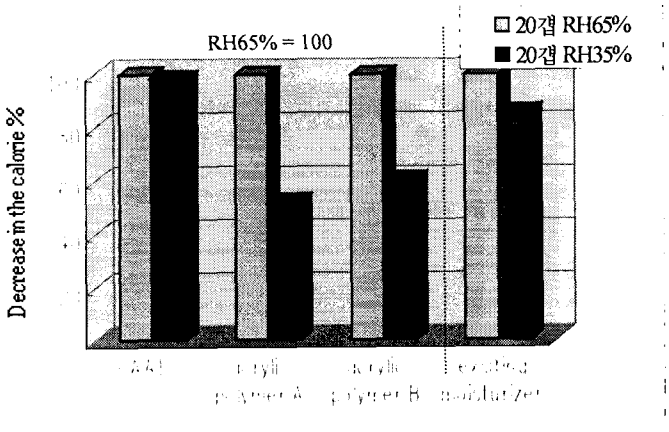


Fig.9 The remaining water content of the polymer-treated-hair under several humidity condition (for 5h.) by Karl Fischer Method.



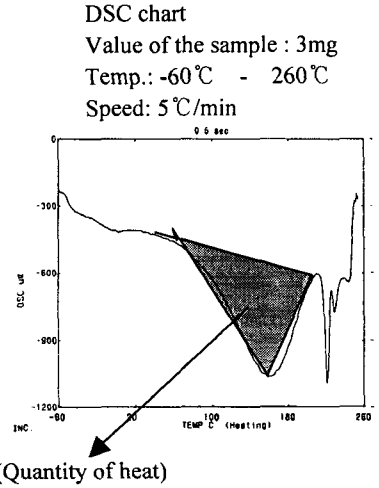


Fig.10 The change in calorie was measured by Differential Scanning Calorimeter (DSC220C SEIKO INSTRUMENTS. INC, Japan) for thermal analysis. We assume that the change in remaining water content corresponds to change in calorie.

CONCLUSION

New formulations which is able to make hair memorize its curl shape, and further more, leaves soft and natural touch feeling on the hair have been developed, based on the thin, homogeneous, and strong film-forming polymer "PAAD". The main chain of this polymer consists of the amino acid, and it is incorporated the various functional groups, like alkyl amide, betaine, to improve the setting effectiveness.

Besides performing as a film-forming polymer, "PAAD", as an active ingredient, has moisturizing effect, just like existing moisturizer.

Moreover, we expect that this polymer has a various features in changing the alkyl length of the side chain and the composition ratio of monomer without changing of the main chain.

We can't only apply this polymer to styling products, but also to hair treatments.

REFERENCES

[1] Woodard, J., J. Soc. Cosmet. Chem., 23, 593 (1972)

[2] S. R. Wendel, A. J. Disapio : Cosmet. Toiletr., 98, 103 (1983)

[3] Toru Simizu, Kiyomi Yokoohji : Fragrance Journal, 12, 22-30 (1993)

[4] Kazuhide Hayama, *et al* : Fragrance Journal, 37, 69-76 (1979)