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GENU pHresh™ DF Pectin Helps Protect the Skin Barrier

Using pectin in skin care:

- Assists the skin to quickly restore and maintain its acid protective barrier.
- Soothes skin after soap cleansing.
- Its moisturizing effect on stratum corneum and protective action against water loss has been clinically demonstrated.
- Pectin is a polysaccharide extracted from citrus peel, a natural source.
- Pectin does not cause irritation.

What is pectin?

Pectin is a natural polysaccharide associated with all superior plant cell walls[1].
GENU pHresh™ DF pectin is a unique proprietary pectin extracted from citrus fruit, in which it provides structure to the protective fruit peel and builds the pulp cells containing the juice.

How does pectin assist the skin in reforming its acid mantle?

Both lipid organization and lipid metabolism in the stratum corneum require an acidic pH. The protective layer formed by sebum at acid pH on the surface of the skin is called acid mantle. Several authors have described the acid mantle of the skin[2,3,4,5,6]. Skin pH varies naturally from pH 4.1 to 5.8, with an average around 4.8. It can be affected by a great number of endogenous factors, e.g. skin moisture, sweat, sebum, anatomic site, genetic disposition, and age. In addition, exogenous factors like detergents, application of cosmetic products, and occlusive dressings may influence the skin pH for several hours.

In skin care, maintaining the natural acid mantle of the stratum corneum is important for both permeability barrier formation and cutaneous antimicrobial defense. Failure to readily regenerate the acid and oily part of the epidermis often results in dermatological symptoms, such as itching, chapping, and cracking of the epidermis, especially during cold weather. It will damage natural microflora and fail to neutralize harmful contaminants, leading to potential infections.

It has been recently documented that skin with pH value below 5.0 is in better condition than skin with pH value above 5.0.
GENU pHresh™ DF pectin works as a weak polymeric acid; it possesses high pH buffering capacity with a pKa around 4, combined with a long term alkali scavenging ability, which helps quickly restore and stabilise cutaneous pH over time.

BACKGROUND INFORMATION ON PECTIN

Pectin is a complex polysaccharide, which consists of an alpha 1-4 linked polygalacturonic acid backbone intervened by rhamnose residues and modified with neutral sugar side chains and non-sugar components such as acetyl, methyl, and ferulic acid groups.

The galacturonic acid residues in pectin are partly esterified and present as the methyl ester. The degree of esterification (DE) is defined as the percentage of carboxyl groups esterified. Pectin with a degree of esterification above 50% is named high methyl ester (HM) pectin or high ester pectin and one with a DE lower than 50% is referred to as low methyl ester (LM) pectin or low ester pectin. Most pectin found in plant material, such as fruits, vegetables, and grass, is HM pectin.

Kertesz[7] showed in 1951 that when alkali was added to a pectin solution in an amount exceeding the amount for neutralizing the pectin, de-esterification occurred. The process consumed alkali, resulting in a pH-drop of the solution. He suggested that the consumption of alkali be used for determining the ester content of pectin.

However, pectin is also a weak organic acid with a pKa value of about 4.

So, pectin is capable of performing the two following basic reactions involving consumption of alkali:

1. **Acid – Base equilibrium, which provides an immediate buffering effect:**

$$R\text{-}COOH + OH^- \rightleftharpoons R\text{-}COO^- + H_2O$$

This reaction is predominant for low DE pectin.

2. **De-esterification, which provides a scavenging effect on alkali over time:**

$$R\text{-}COOCH_3 + OH^- \longrightarrow R\text{-}COO^- + CH_3OH$$

This reaction is predominant for high DE pectin.

To prove the effect of pectin on skin acid mantle, CP Kelco performed a series of tests, from in vitro evaluation of buffering and the scavenging effect of pectin, to in vivo clinical tests to demonstrate its performance to protect skin barrier functions and moisturization ability of the stratum corneum.

IN VITRO EVIDENCE OF BUFFERING EFFECT OF PECTIN

CP Kelco performed a series of trials to demonstrate pectin's ability to consume alkali and drop pH:

- Determination of immediate alkali consumption (buffering effect):
  - A 2% pectin solution was placed in a water bath at 25°C, and titrated with 0.1 M NaOH.
- Determination of pH-drop over time (scavenging effect):
  - A simple test was designed, where the pH of a 1% pectin solution was added 0.1 M NaOH to a pH of 10. At a constant temperature, the pH-drop over time was recorded.

Results and Discussion

If we consider the pH drop of a solution, a pectin will have an immediate effect on the pH, being an acid. This means that it will be capable of maintaining a relative constant pH over a relatively large range of alkali concentrations.

If we consider the long term action of alkali consumption and scavenging effect, it will be time dependent.

The effect on the buffer effect and the rate of pH drop of the degree of esterification is pictured in **Figure 1**.

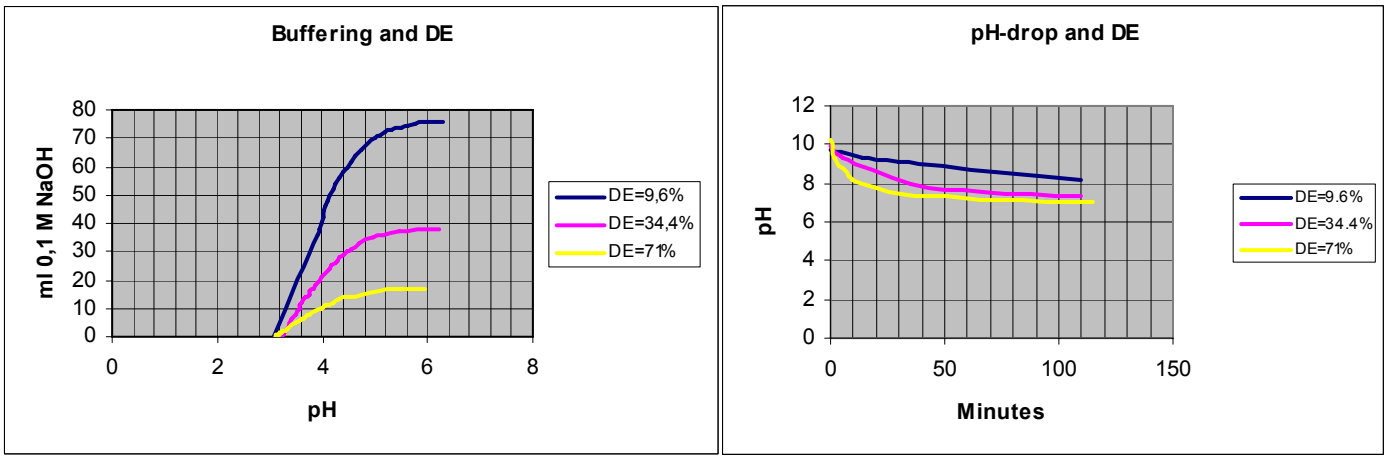


Figure 1: Effect of degree of esterification of pectin on buffer capacity and rate of pH-drop

It is evident that as the degree of esterification increases, the ability of pectin to consume alkali decreases. A low DE pectin contains more carboxylic acid groups and has a more immediate effect on alkali consumption.

Contrary to this immediate buffering effect, the scavenging reaction goes faster as the degree of esterification of pectin increases. If we consider the time needed to drop a solution from pH 10 down to pH 8, a high DE pectin will have a quicker action than a low DE pectin: 12 minutes for a DE of 71%, 35 minutes for a DE=34.4%, 130 minutes for a DE=9.6%. The difference is even bigger to reach a pH of 7.

Therefore, by combining high DE and low DE pectin, one can achieve both immediate alkali consumption and a time-dependant removal of alkali once the buffer effect of the low DE pectin has been depleted. **GENU pHresh™ DF** pectin is the optimised version with the right concentration of high and low DE of pectin for lotions, with both quick and long term effects on the pH buffering.

If we extrapolate this action to skin buffering capacity, the low DE part of **GENU pHresh™ DF** pectin will have an immediate action on pH drop after soap cleansing, the high DE part will have a sustainable action over time on the skin.

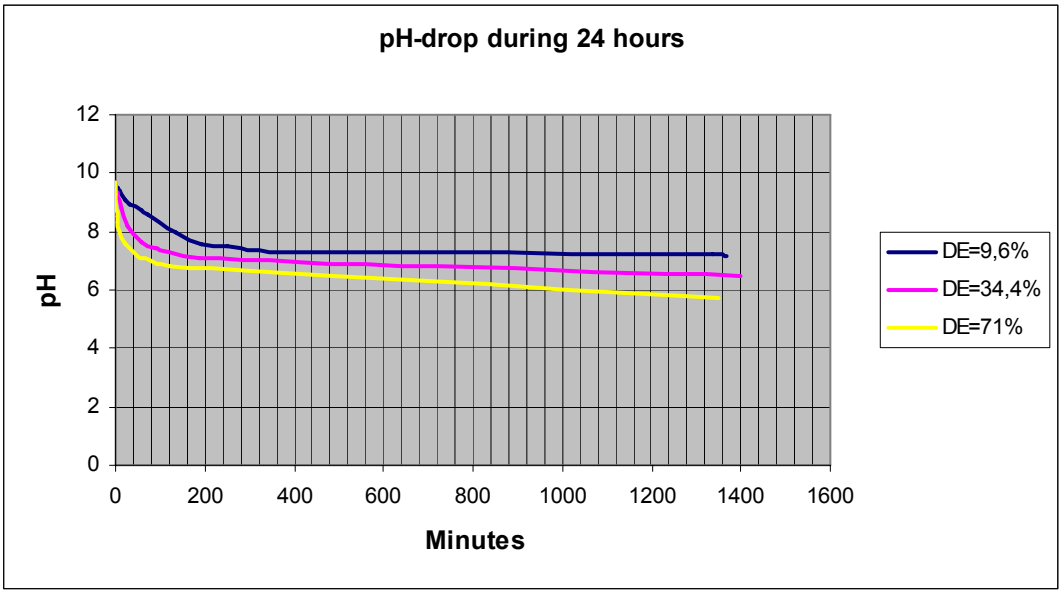


Figure 2: Effect of degree of esterification of pectin and rate of pH-drop over long period of time

Figure 2 shows the pH-drop over a long period of time of three pectin preparations at 30 – 32°C. One preparation contains pectin with a DE of 9.6%, another contains a pectin with a DE of 34.4%, and the third – a pectin with a DE of 71%.

Going further in this demonstration, the ability of a high DE pectin to regenerate after several cycles of alkalinisation was tested, as a simulation of potential alkali aggressions of the skin during the day (skin pH can be increased by simply watering the skin or basic contamination by pollutants present in the atmosphere).

Figure 3 shows the effect of multiple dosages of alkali. The pH drop for one sample having DE=71% was recorded at a temperature of 25 – 27°C. First, the pH was raised to about 10 with 19 ml. 0.1 M NaOH. When the sample had reached a pH of 6 – 7, the pH was again raised to about 10. This required 1.1 ml. 0.1 M NaOH. When the pH had reached 6 – 7, the pH was raised a third time to about 10, which required 1.2 ml 0.1 M NaOH.

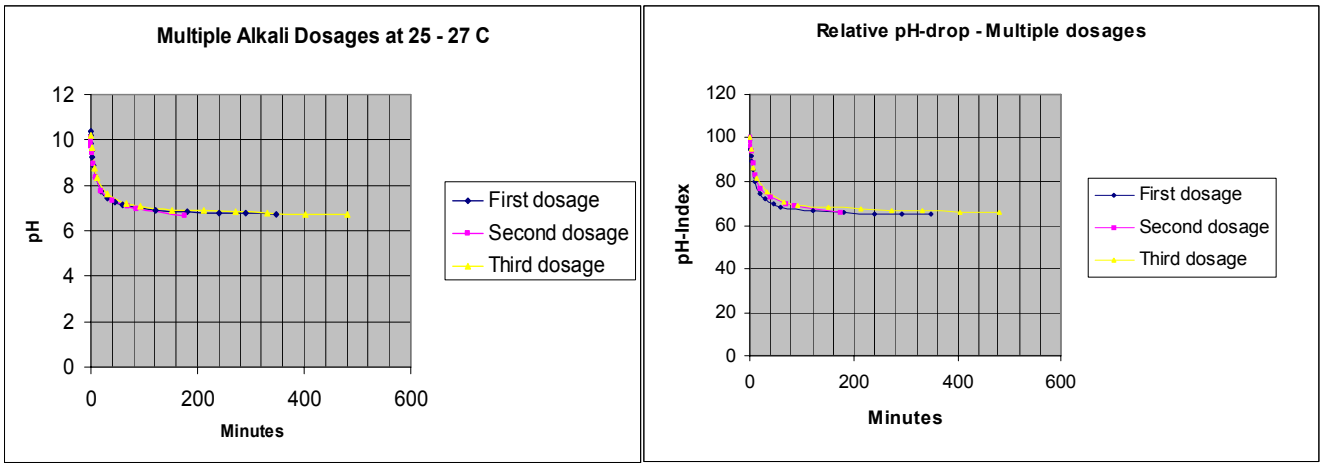


Figure 3: Effect on rate of pH-drop of multiple alkalinity cycles

The absolute results show that the rate of the pH-drop stays unchanged after at least three cycles, where the pH is first increased to about 10, then after the pH has dropped increased to about 10 again. However, when plotting the pH-index as a function of the rate of pH-drop, it becomes clear that the rate is decreased after each cycle. After one cycle, the DE is decreased to about 66%, so the ability to continue reducing pH is caused by an incomplete de-esterification.

Still, if alkalinity appears in pulses, for at least three times pectin is able to reduce the alkali. In fact, in one experiment, which went on for seven days, a 200 ml 1% pectin solution of DE=71% consumed 73 ml. of a 0.1 M NaOH solution. After this period, the DE had decreased to 9.1%.

This demonstrates the ability of pectin to maintain low pH over time after several cycles of alkali addition. GENU pHresh™ DF pectin will help the skin to quickly regenerate its natural pH during the day when it is affected by different actions from endogenous or exogenous factors.

Optimal use levels of GENU pHresh™ DF pectin:

Complementary tests showed as well that the molecular weight of pectin does not influence the buffer effect or the rate of pH-drop, which in practice means that the buffer effect and the rate of the pH-drop can be accomplished with or without a substantial viscosity increase of the preparation.

As well, tests on pectin concentrations in a lotion showed that buffering effect will increase together with pectin use level and will start to be significant above 1% of pectin in the water phase.

We recommend a use level of 1.25% GENU pHresh™ DF pectin in lotions and creams for optimal effect.

CLINICAL TESTING

I. Repeat Insult Patch Testing

Pectin is already used in cosmetic and skin care application and has an INCI name. CP Kelco had it dermatologist-tested by an independent outside laboratory: BioScreen Clinical Services, 3892 Del Arno Blvd, Torrance, CA 90503, USA by the Repeat Insult Patch Test (RIPT).

The testing was done on 51 subjects. Approximately 0.05 ml of 1% to 2% pectin solution was placed on a 7 mm disc. The occlusive test discs were taped to the subjects' backs for 2 days and then evaluated. This procedure was repeated for nine consecutive two-day exposures, three times a week for three weeks. After a 10 to 14-day rest period, a retest dose was applied to a previously unexposed site to assess sensitization.

Results: None of the test material caused significant irritation or sensitization under the conditions it was applied. No glazing, peeling, hypopigmentation, hyperpigmentation, or fissuring was observed. No adverse reactions were noted. None of the products caused contact sensitization (allergy), confirming other research from Rheins[8,9].

Although pectin is a fruit acid, it does not cause skin irritation or skin sensitization. Other acids, such as alpha hydroxy acid, are known to cause irritation of the skin. The fact that pectin does not show this effect is believed to be due to pectin being a polymer, which is unable to penetrate the skin. Likewise, pectin does not cause peeling of the skin.

II. Evaluation of Buffering Capacity and Moisturization Potential of the Stratum Corneum

CP Kelco had a high metoxyl pectin dermatologist-tested by an independent outside laboratory: BioScreen Clinical Services, 3892 Del Arno Blvd, Torrance, CA 90503, USA, to evaluate its role to buffer skin pH and to provide a Moisturization Effect to the Stratum Corneum.
A 1% pectin solution, pH 3.36, was tested as a rinse after soap cleansing on the forearms of 29 subjects, with various age range, sex, and race.
Seven days prior to the start of the study, the subjects began a «washed-out» period using a bar of Neutrogena fragrance free facial bar. This insured that all the subjects' skin was acclimated prior to any bioinstrumental assessment.

Three control measurements were used:

- Evaporative water loss from the skin correlates to the degree of perturbation of the skin. An insulated probe (DermaLab TEWL device, Cortex Technology, Hadsund, Denmark) with a sensor was gently applied to the surface of the forearms and a gradient of relative humidity was measured. Three measurements were obtained at each TEWL reading, at baseline, following the soap wash, and following the one hour skin temperature, relative humidity reequilibration.
- Skin capacitance was evaluated with a Corneometer CM825 (Courage-Khazaka, Kohn, Germany). It measures dielectric constant occuring in the stratum corneum. Increasing the dielectric constant leads to decreased impedence and increased conductance and capacitance. The higher the capacitance value the more hydrated the stratum corneum corneocytes. Three measurements were obtained for both the treated and untreated control sites, at baseline, following the soap wash, and following the one hour skin temperature, relative humidity reequilibration.
- The pH of the treated and controlled sites were obtained by using an Orion 3 Star pH meter (Thermo-Corporation, Beverly, MA), with a flat pH probe. Three separate pH measurements were obtained, at baseline, following the soap wash, and following the one hour skin temperature, relative humidity reequilibration.

The subjects were washed for 40 seconds with Colgate Irish Spring soap, a sodium salt of fatty acids, rinsed with deionized water and patted dry. The three measurements were repeated in triplicate.
2 ml of the test pectin were applied to one forearm on each subject. The other site was treated with 2 ml of water. After 60 minutes at 30% relative humidity and 20-22°C, the three measurements were repeated in triplicate.

Results:

Difference = TreatChange (PT–B2) – ContChange (PT–B2)
Where: PT = post treatment, B2 = baseline 2 after soap exposure of the skin sites, Treat = treatment, Cont = Control.

TEWL:

Variable	Mean	N	Minimum	Maximum	Pr > [t]
Avg Control	3.025	29	-6.0	16.7	
Avg PostControl	-2.953	28	-14.9	3.6	
Avg Treat	3.106	28	-4.9	20.7	
Avg PostTreat	3.204	29	-20.4	3.5	
Change	0.249	28	-12.3	8	0.6128

Corneometer:

Variable	Mean	N	Minimum	Maximum	Pr > [t]
Avg Control	9.560	29	-6.100	27.400	
Avg PostControl	-9.225	29	-22.200	3.367	
Avg Treat	9.079	29	-3.300	34.467	
Avg PostTreat	-13.274	29	-38.400	1.633	
Change	-4.048	29	-29.567	13.000	0.0177

pH:

Variable	Mean	N	Minimum	Maximum	Pr > [t]
Avg Control	1.402	29	-0.163	2.953	
Avg PostControl	-0.473	29	-1.417	0.857	
Avg Treat	0.968	29	-0.627	3.297	
Avg PostTreat	-1.528	27	-3.353	-0.170	
Change	-1.064	27	-3.350	0.573	< .0001

Conclusions:

Under the conditions of the study, the pectin solution demonstrated:

- a significant (p<.017) increase in stratum corneum moisturization utilising the CM825 Corneometer
- a directional increase in transepidermal moisturization, following DermaLab TEWL measurements
- a highly significant (p<.0001) pH buffering capacity following pectin treatment versus the deionized water control

III. Long Term Protective Effect on Skin Barrier

An in-use test was performed to evaluate and compare the efficacy of a lotion containing pectin to a placebo lotion, after repeated applications on half-face for 4 weeks, under normal conditions of use, in the female adult subject, at an external independant laboratory: Institut d'Expertise Clinique, 88 bd des Belges, 69006 Lyon, France.
A basic lotion containing **GENU pHresh™ DF** pectin, blend of HM and LM pectin, and a placebo lotion were applied twice a day during 28 days after cleansing on half face of 20 subjects with dry or very dry « hypersensitive » face skin (selected by the stringing test method).

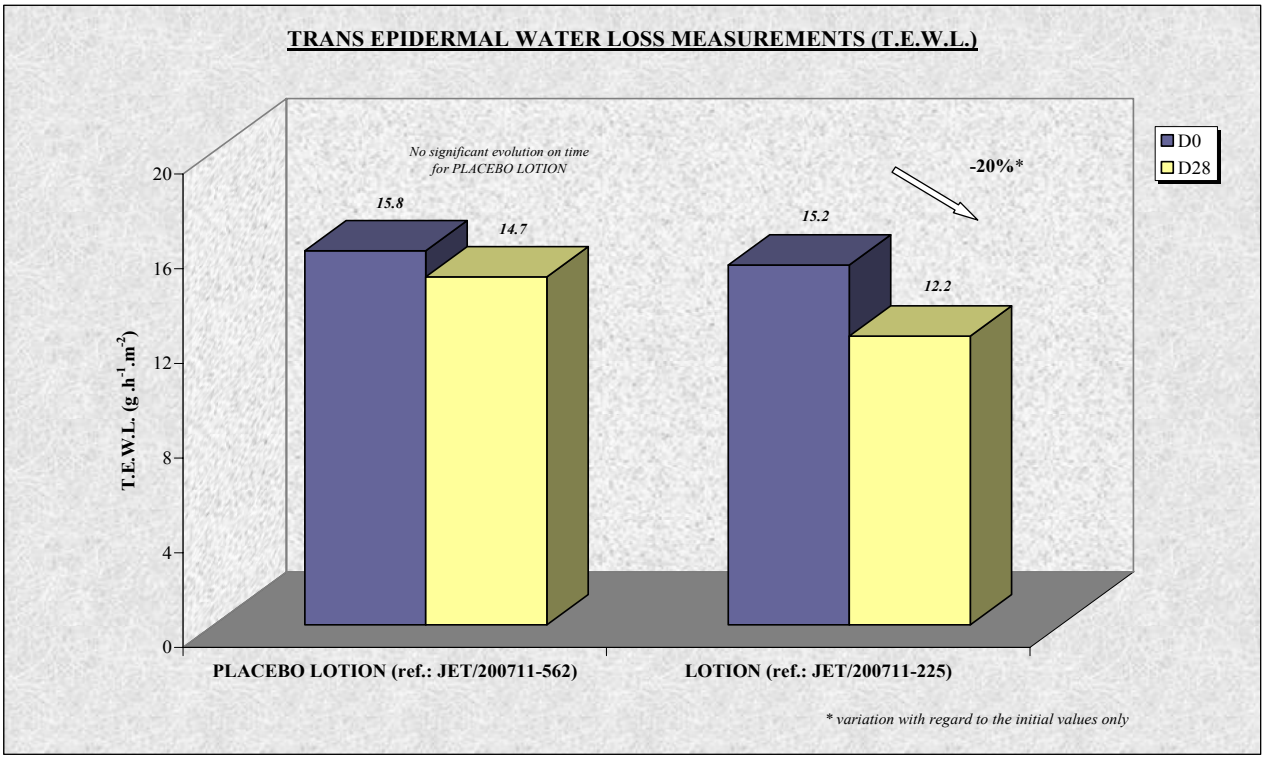
- Transepidermial waterloss was measured at baseline D0 and after 28 days D28, using a Tewameter TM300 (Courage + Khazaka, Germany).
- Self-assessment questionnaires with closed questions on efficacy and cosmetic qualities of each investigational product were filled out by the subjects.

Lotion Formulations:

Ingredients	Placebo Lotion	Pectin Lotion
GENU pHresh™ DF pectin	0.00	1.15
Isopropyl Palmitate	18.00	18.00
Emulsifier	6.10	6.10
Sodium benzoate	0.07	0.07
Potassium sorbate	0.05	0.05
Distilled water	75.78	74.63
Total	100.00	100.00

Results:

Figure 4 illustrates the effect of lotion containing pectin on skin with a statistically significant decrease of 20% in the Transepidermal Waterloss on the area treated with the lotion, in comparison with the initial values, and in comparison with the placebo lotion, showing a « protective effect against water loss » and an « improvement of the skin barrier function », as described in French Regulation for cosmetic product efficacy evaluation (DGCCRF December 1994).



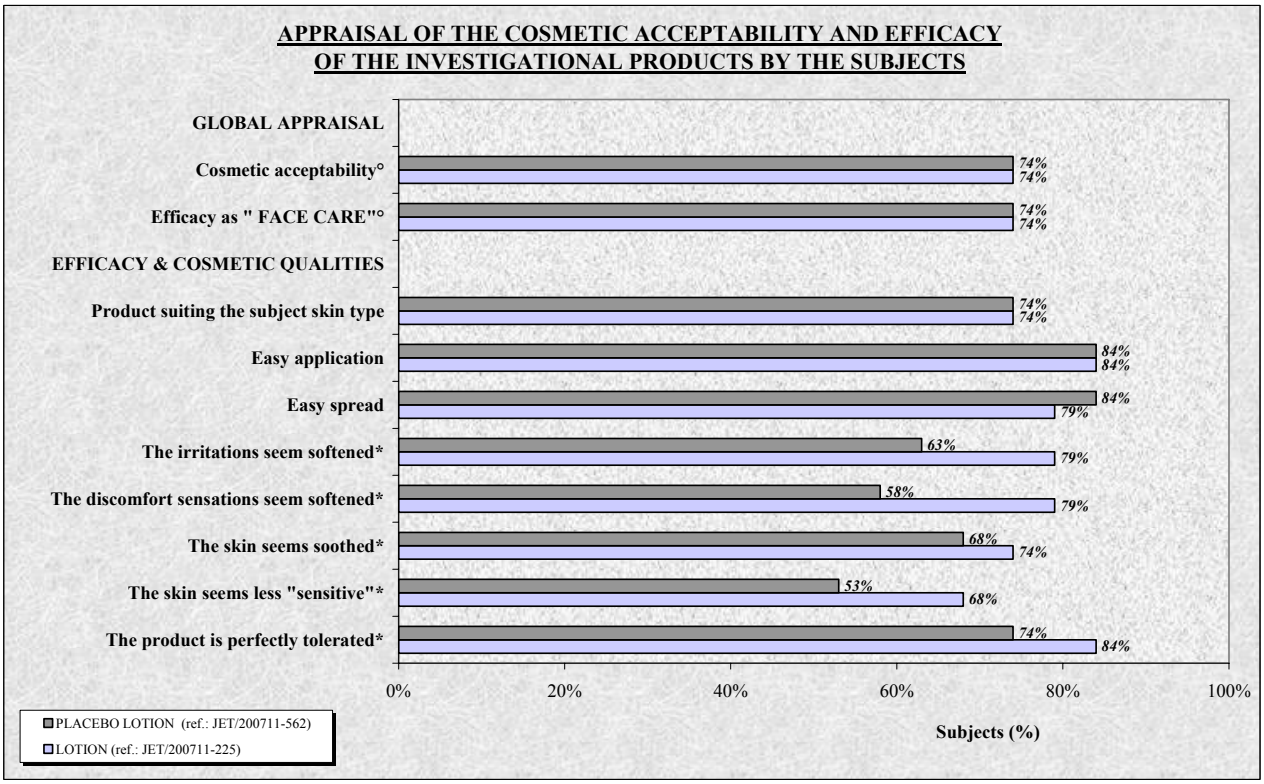


Figure 4: TEWL measurement and appraisal by subjects of pectin lotion compared to placebo lotion after 28-day study

IV. In Vivo Evolution on Skin pH After Soap Cleansing

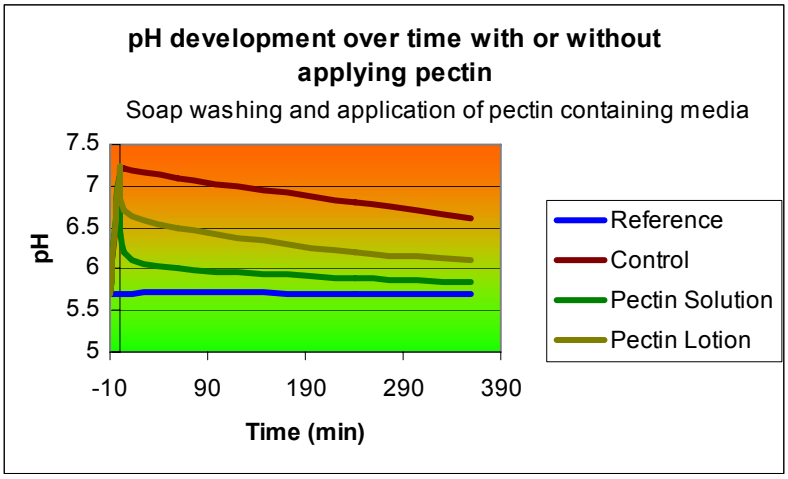


Figure 5: Combined results of different in vivo pH measurements over time, showing the buffering effect of pectin on skin pH.

FORMULATIONS AND APPLICATIONS FOR GENU pHresh™ DF PECTIN

Skin Cream Formulation—Post-Shave / Post-Depilatory Cream

Ingredients	QTY(%W/W)
Water	q.s. to 100
GENU pHresh™ DF pectin	1.25
Glycerin	2.0
Cetyl alcohol (and) Glyceryl Stearate (and) PEG-75 Stearate (and) Ceteth-20 (and) Steareth-20	6.0
Dimethicone	1.0
Cetyl octanoate	3.0
Mangifera Indica (Mango) seed butter	1.0
Caprylic capric tryglyceride	8.0
Cetearyl alcohol	2.0
Tocopheryl acetate	0.3
Macadamia Integrifolia seed oil	2.7
Preservative	q.s.

pH:	4- 5.00
Stability:	Stable for at least 1 month @45°C

PROCEDURE:

Disperse **GENU pHresh™ DF** pectin in the water phase under high speed mixing. Add glycerin.
Heat Phase A until 80-85°C, mix to hydrate pectin. Heat Phase B until 75-80°C.
Add Phase B with propeller mixing to Phase A. Homogenize for a short time (2500 rpm for 1 min.).
Start cooling with mixing.
Add remaining ingredients below 45°C.
Mix to get a uniform cream. Adjust water loss.

Notes on formulating a cream or a lotion with pectin

- ✓ Other components of the lotions/creams should be stable in acid conditions.
- ✓ To provide for improved stability of the lotions/creams at elevated temperatures (45°C), it is recommended to use non-ionic stabilizers, which do not interact with pectin. Examples of non-ionic stabilizers include guar gum, locust bean gum and hydroxyl ethyl cellulose. A particularly useful stabilizer is refined locust bean gum, which produces the smoothest lotion. However, when using locust bean gum, it is recommended to heat the water phase with the locust bean gum to 85°C in order to ensure complete dissolution of the stabilizer.
- ✓ Pectin can be added to either the water phase or the oil phase of the lotions/creams. Since pectin is insoluble in the oil phase, the undissolved pectin particles will tend sink to the bottom, and stirring must be maintained until the addition of water phase.

Potential Applications

The concept of using pectin as an alkali scavenger could potentially be utilized in a range of products being in contact with skin to assist the skin in reforming its acid mantle.
Such products include:

Facial and body lotions and creams - product ranges for sensitive skin, acne (soothing effect and restores pH of natural flora against outside contaminants), suncare, after depilatory, peeling, whitening treatments.

Shaving products - The application of a conventional shaving preparation of high alkalinity is often irritating to the skin[10]. Pectin could be useful to provide for a non-irritating skin pH.

Deodorant products - The apocrine glands secrete a milky fluid that has a pH range of 5 to 6.5 and initially consists of lipids, proteins, and carbohydrates[11].

Hair care - hair tonics/serums to normalize pH on scalp. It has been observed that low pH is useful in reducing dandruff, seborrheic dermatitis, and psoriasis. Pectin can not be used in hair care formulations containing cationics.

Liquid soaps

Feminine hygiene

Pro-fragrance products - compounds, which under certain conditions break down to fragrances. For instance, tris(9-decenyl) when exposed to suitable conditions (e.g., exposure to the acid mantle of human skin) breaks down to release a mixture of 9-decenol and 9-decenyl formate, both of which are fragrance raw materials[12,13,14]. However, if the skin pH is too high, the hydrolysis is too slow, and thus, the use of pectin together with the pro-fragrance may ensure the right pH for development of the fragrance.

Lotionized tissue products - While products exist to address the problems of skin irritation and inflammation, they inevitably fail to address the short-term impact of various additives on the pH balance of the skin, i.e., the skin's acid mantle. To put this into perspective, one need only to consider conventional facial tissue, toilet tissue, napkin and paper towel products that are used for wiping dry or wet skin. Upon contact with skin, the tissue products transfer some of the chemicals present in the tissue to the skin surface[15].

Fabric rinses

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Further Information

More detailed information on pectin's ability to affect pH is available in:

Trudsø, J.E.: Pectin is an Alkali Scavenger, CPKelco ApS, 2006

Trudsø, J.E.: SKIN-PROTECTING ALKALINITY-CONTROLLING COMPOSITION AND THE USE THEREOF, WO 2005102262, filed April 26, 2004

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