

Phytochemical and thickening properties of the mucilage of *Malachra alceifolia* jacq., in a shampoo formulation

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Propiedades fitoquímicas y espesantes del mucílago de Malachra alceifolia jacq., en una formulación de champú

Propietats fitoquímiques i espessidors del mucílago de Malachra alceifolia jacq., en una formulació de xampú

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ABSTRACT

The feasibility of the using of mucilages as viscous hydrocolloids in order to reduce dependence on inorganic products that are unfriendly to the environment, is a necessity that the scientific community must take advantage on behalf of development of new products. The aim was to determine the phytochemical components and thickening capacity of mucilage as a hydrocolloid in a shampoo formula. Phytochemical sieve was done by coloration, precipitation and flotation reactions. Mucilage extraction was performed by hydration-maceration of crushed stems according to experimental design 2³ (three factors and two levels) and controlled by the viscosity response variable. For the shampoo, a design of 2 was used (two factors and two levels), evaluated with viscosity response variables and lather formation, results that were tabulated with the statistical software statgraphics plus. The results show in the hydrocolloid the presence of tannins, alkaloids, triterpenes. Its thickening capacity due to the significant viscosity of the hydrocolloid (1186 mPa.s). The viscosity of the shampoo between 1020 and 1460 mPa.s and the amount of foam (213 ml) that reward

the quality. It is concluded that, the complex molecular structure and abundant presence of mucilage in its natural state in the stems of *Malachra alceifolia* Jacq allows the hydrocolloid obtained to have a great viscosifying and moisturizing capacity with an almost neutral pH, qualities that affect the quality of the shampoo and are suitable for commercial products with friendly features to the environment; being promoted as a raw material for formulations of other personal hygiene products with an important projection of immediate application in the pharmaceutical and cosmetic industry.

Keywords: mucilage, viscosity, shampoo, bioproduct

RESUMEN

La factibilidad del uso de mucílagos como hidrocoloides viscosos para reducir la dependencia de productos inorgánicos nocivos para el medio ambiente, es una necesidad que la comunidad científica debe aprovechar en pro del desarrollo de nuevos productos. El objetivo fue determinar los componentes fitoquímicos y la capacidad espesante del mucílago como hidrocoloide



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en la fórmula de un champú. El tamiz fitoquímico se realizó mediante reacciones de coloración, precipitación y flotación. La extracción del mucílago se realizó por hidratación-maceración de tallos triturados según diseño experimental 2³ (tres factores y dos niveles) y controlado por la variable respuesta viscosidad. Para el champú se utilizó un diseño de 2² (dos factores y dos niveles), evaluado con variables de respuesta de viscosidad y formación de espuma, resultados que fueron tabulados con el software estadístico statgraphics plus. Los resultados muestran en el hidrocóloide la presencia de taninos, alcaloides, triterpenos. Su capacidad espesante se debe a la importante viscosidad del hidrocóloide (1186 mPa.s). La viscosidad del champú entre 1020 y 1460 mPa.s y la cantidad de espuma (213 ml) que premian la calidad. Se concluye que, la compleja estructura molecular y abundante presencia de mucílago en estado natural en los tallos de *Malachra alceifolia* Jacq permite que el hidrocóloide obtenido tenga una gran capacidad viscosa e hidratante con un pH casi neutro, cualidades que inciden en la calidad del producto. champú y son aptos para productos comerciales con características amigables con el medio ambiente; siendo promovida como materia prima para formulaciones de otros productos de higiene personal con una importante proyección de aplicación inmediata en la industria farmacéutica y cosmética.

Palabras clave: mucílago, viscosidad, champú, bioproducto

RESUM:

La viabilidad de l'ús de mucílags com a hidrocol·loides viscosos per tal de reduir la dependència de productes inorgànics no respectuosos amb el medi ambient, és una necessitat que la comunitat científica ha d'aprofitar per al desenvolupament de nous productes. L'objectiu era determinar els components fitoquímics i la capacitat d'espessiment del mucílago com a hidrocol·loide en una fórmula de xampú. El tamís fitoquímico es va fer mitjançant reaccions de coloració, precipitació i flotació. L'extracció de mucílago es va realitzar per hidratació-maceració de tiges triturades segons el disseny experimental 2³ (tres factors i dos nivells) i controlada per la variable resposta de viscositat. Per al xampú es va utilitzar un disseny de 2² (dos factors i dos nivells), avaluat amb variables de resposta de viscositat i formació d'escuma, resultats que es van tabular amb el programari estadístic statgraphics plus. Els resultats mostren en l'hidrocol·loide la presència de tanins, alcaloides, triterpens. La seva capacitat espesidora a causa de la important viscositat de l'hidrocol·loide (1186 mPa.s). La viscositat del xampú entre 1020 i 1460 mPa.s i la quantitat d'escuma (213 ml) que premien la qualitat. Es conclou que, la complexa estructura molecular i la presència abundant de mucílago en estat natural a les tiges de *Malachra alceifolia* Jacq permet que l'hidrocol·loide obtingut tingui una gran capacitat viscosificant i hidratant amb un pH gairebé neutre, qualitats que afecten la qualitat del xampú i són adequats per a productes

comercials amb característiques favorables al medi ambient; impulsant-se com a matèria primera per a formulacions d'altres productes d'higiene personal amb una important projecció d'aplicació immediata en la indústria farmacèutica i cosmètica.

Paraules clau: mucílago, viscositat, xampú, bioproducte

INTRODUCTION

The genus *Malva L.* (Malvaceae) is represented by 40 taxa worldwide¹, México 75 species². It is a typical plant of the Mediterranean region; it contains 240 genera and more than 4200 species³. Plant whose base and stem is a bit woody known as Malva de las laderas⁴. *Malachra alceifolia* Jacq, known as Malva, Malva de Caballo or Borage has a high mucilage content⁵. The mucilage is found in the flowers⁶. and mostly in the pith of the Malva peruviana stems, which when extracted form hydrocolloids with a great capacity to bind impurities⁷. The term hydrocolloid is used to describe a variety of polysaccharides and proteins from tree, plant, algae, microorganism, and animal sources to improve the viscosity, texture, and stability of foods⁸.

The mucilage in Malvaceae is produced within parenchymal cells by hydrolysis of the cellulose wall (change from cellulose to hydrocellulose); when it is hydrated, it swells and compresses the protoplasm towards the center of the cell, giving rise to radiant threads that imitate the threads within the protoplast extended from the nucleus to the wall layer⁹. It shows lamination (layers opposite the cell wall in the form of mucilage threads) in the mucilaginous ducts, determined by the water content, which generates the idea that the mucilage arises as a secondary thickening in the structural sense¹⁰. Normal natural products of cellular activity¹¹. They are soluble fibers and as vegetable hydrocolloids they arouse great interest at a therapeutic level and within pharmacology, pharmaceutical technology¹², and cosmetic.

Mucilages are polysaccharides, in water they form viscous colloidal solutions with a high capacity to hydrate¹³, insoluble in water and coagulate in alcohol¹⁴⁻¹⁵. They constitute a diverse class of macromolecules in high concentrations in different parts of the plants¹⁶. Absorb water up to 5 times their mass, forming thick and viscous solutions that appear as a time-dependent non-Newtonian fluid¹⁷. They are polysaccharides with a physicochemical diversity and different structure, which have functional and health benefits¹⁸.

Mucilage, polysaccharide that is mainly composed of carbohydrates with highly branched structures consisting of monomeric units of L-arabinose, D-xylose, D-galactose, L-rhamnose and galacturonic acid¹⁹. Its application in the pharmaceutical and cosmetic industries is mainly related to hemicelluloses in its composition, except for the sugars produced by hemicelluloses such as xylose, glucose and mannose²⁰. Mucilages have a slightly acidic pH²¹, present in certain plant species in different parts of the plant (root, tubers, peel, inner of stems, leaves, flowers and seeds) used as refreshing, moisturizing and thickening agents.

Malachra alceifolia used as emollient, moisturizing for the skin and for infections and anti-inflammatory²², anti-inflammatory, emollient, stimulant, galactogenic²³, important for cosmetic products. Aqueous colloidal dispersions “moisturise and protect the skin surface”²⁴. The use of mucilages in the formulation of cleaning products is guided by its components (phytochemical sieve) that value the biological and pharmacological potential of the plant²⁵.

Natural herbal shampoos, are becoming more and more popular²⁶, and the trend is the use of 100% natural products²⁷; that are soft, clean, with a pleasant aroma, with the capability to untangle the hair and that generate characteristic shine. The shampoo is the most common form of hair treatment, most of which are neutral or slightly acidic²⁸, with pH less than or greater than 5.5²⁹. Slightly acidic pH and viscosity in the range between 1250 to 9000 cP³⁰, in addition to lather capacity and color, they are variables that are ensured with the addition of mucilage in the formulation of the shampoo, avoiding sodium chloride, a substance commonly added as a thickener to synthetic shampoos. The aim of the study was to determine the phytochemical components and thickening capacity of mucilage as a hydrocolloid in a shampoo formula. Those are properties that give the obtained product from the stems of *Malachra alceifolia* Jacq that are favorable qualities that make it a scientific novelty to develop new technologies in the production of personal hygiene bioproducts.

MATERIALS AND METHODS

The plant species *Malachra alceifolia* Jacq, was collected in stubble and pastures on the coast at 50 meters of altitude. Plants up to 2.5 m tall were collected, with intense yellow flowers, leaves with hispid hairs and stems rich in mucilage stored in the pith of the stem “layers which are opposite to the cell wall in the form of mucilage strands in the mucilaginous ducts”³¹, and that, when crushing and hydrating them in water (maceration-soaking), it generates a viscous

and thick product (hydrocolloid) which transferred to another container, the slime does not separate or cut easily (figure 1) and, for the control of variables, it was used an experimental design 2³ (table 1) with agitation for 2 minutes.

To identify secondary metabolites (alkaloids, tannins, saponins, flavonoids, triterpenes, steroids, acid-base quinones, quinones (α β), carotenoids, coumarins and naphthaquinones), Phytochemical sieve was performed using hydrated natural mucilage (hydrocolloid) as a sample by rapid reactions.

Table 1. Experimental design applied in the extraction of the mucilage

Factors	Units	Levels		Variable response
		Low	High	
Concentration (X1)	g/L	80	160	Viscosity (mPa.s)
Maceration time (X2)	min.	30	60	
Stirring speed (X3)	rpm	100	180	

To make one kilogram of shampoo (commercial recipe book) was used: Cooperland (32 g), cethiol (10 g), lanolin (5 g), vitamin E and preservative (6 g), “14% \pm 1 texapon-sodium lauryl ether sulfate”²⁷ and rest of hydrocolloid (g) at a concentration of 160 g/L and the rest of malva hydrocolloid obtained at a concentration of 160 g/L, according to experimental design 2² (table 2).

Table 2. Experimental design used in the preparation of shampoo

Factors	Units	Levels		Variable response
		Low	High	
Amount of Mucilage (X1)	g	790	840	Viscosity (mPa.s)
Texapone-Sodium Lauryl Ether Sulfate (X2)	g	105	158	Amount of foam (ml)

To measure the dynamic viscosity of the mucilage and the shampoo, a CGOLDENWALL NDJ-5S rotational

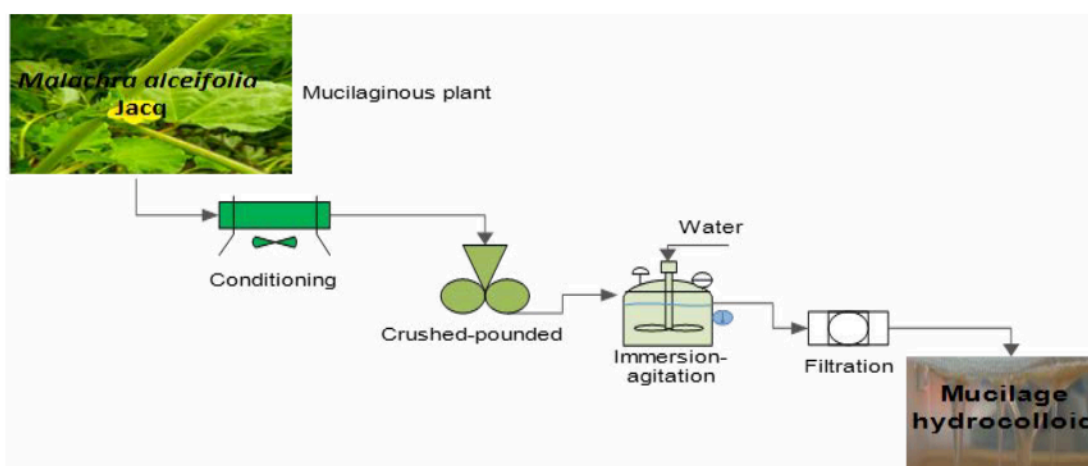


Figure 1. Process for obtaining mucilage by hydration.

viscometer was used, measurement range between 10 mPa.s-100,000 mPa.s, performed with spindle # 1, at 6 rpm and temperature control (18°C ±1). For measuring the amount of foam, the 1000 ml capacity test tube technique was used (2 grams of shampoo in 100 ml of tap water in solution), resting for one minute and then stirring for 30 cycles; results that were evaluated using the statistical software statgraphics plus.

RESULTS AND DISCUSSION

Phytochemical analysis

Results of the phytochemical screening (table 3) of the mucilage as a hydrocolloid showed the existence of alkaloids, tannins, triterpenes and saponins (±); which indicate its potential biological and pharmacological use²⁵. Alkaloids with diverse biological activity and tannins have different pharmacological effects such as antioxidant, antimicrobial and anti-inflammatory with dermatological effects³².

Table 3. Phytochemical screening of mucilage hydrocolloid

Parámetro	Resultado	
Alcaloides	(+)	
Taninos	(+)	
Saponinas	(-)	
Flavonoides	(-)	
Triterpenos	(+)	
Esteroides	(-)	
Quinonas	Ácido	(-)
	Base	(-)
Quinonas α, β hidrolizadas	(-)	
Carotenoides	(-)	
Cumarinas	(-)	
Naftoquinonas	(-)	

(+): positivo, (-) negativo, (-+) No específico

Tannins are antimicrobial associated with their antioxidant capacity³³, triterpenes (30 carbon atoms) also have antiseptic and anti-inflammatory properties³⁴, important in toilet products. *Malva sp.* has a wide variety of chemical constituents (such as polysaccharides, coumarins, flavonoids, polyphenols, vitamins, terpenes and tannins) that are found in different plant organs, especially in leaves and flowers, related to its biological activity³.

Mucilage viscosity

In table 4, viscosity is displayed (μ) of the mucilage as a hydrocolloid shows values between 247 to 394 mPa.s for concentrations of 80 g/L (low level) and from 960 to 1186 mPa.s for concentrations of 160 g/L (high level), measured at a material temperature of 19.3±0.5°C and strain angles greater than two digits, behaving as a non-Newtonian fluid, where the viscosity decreases with the increase of strain ratio (table 3). Studies revealed that the apparent viscosity (Pa.s) at shear rate decreases from (3010 mPa.s to 130 mPa.s) when the temperature increases from 5 to 85 °C³⁵.

Table 4. *Malachra alceifolia* hydrocolloid viscosity

BLOQUE	Concentra- tion (X ₁)	Maceration time (X ₁)	Stirring speed (X ₂)	Viscosity (μ)
	g/L	min.	rpm	mPa.s
1	-1	-1	-1	247
1	-1	1	-1	305
1	1	-1	1	1129
1	1	1	1	1186
1	-1	-1	1	281
1	1	1	-1	1170
1	1	-1	-1	960
1	-1	1	1	394
2	-1	-1	-1	245
2	-1	1	-1	308
2	1	-1	1	1126
2	1	1	1	1185
2	-1	-1	1	278
2	1	1	-1	1169
2	1	-1	-1	861
2	-1	1	1	396

The optimization of multiple responses determines in row 4 a viscosity of 1186 mPa.s, achieving a predicted desirability of 0.999 and an observed desirability of 1, whose goal is to maximize in an adequate combination of factors, to optimize the responses in experiments and process.

According to the factors (figure 2), shows significance for the concentration that decreases drastically for the maceration time and agitation speed at high level.

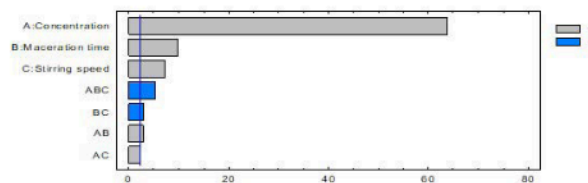


Figure 2. Pareto for viscosity of *Malachra alceifolia* Jacq. mucilage hydrocolloid

For the interactions figure 3, (concentration-maceration time-agitation speed) and interaction (maceration time-agitation speed) the significance is evident at a low level and for the interaction (concentration-agitation speed) it is significant at high level; therefore, the viscosity depends on these factors in their order and in a process, it must be adjusted for adequate optimization in obtaining the hydrocolloid.

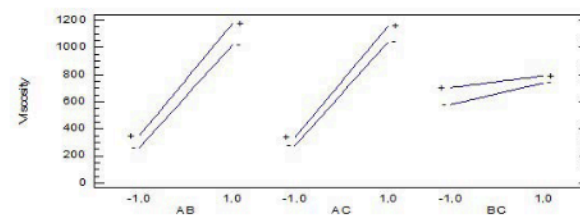


Figure 3. Interactions for Viscosity of *Malachra alceifolia* Jacq. mucilage hydrocolloid

The response surface diagram for viscosity (figure 4) shows that the behavior of viscosity is evident as the concentration increases.

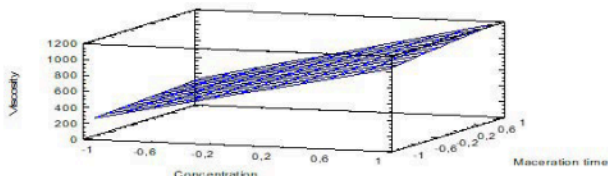


Figure 4. Response Surface for Hydrocolloid Viscosity

Due to the experimentation and according to the significance of the coefficients, the mathematical model, equation 1 (Viscosity, μ) was obtained, which according to the regression coefficient reached a constant in viscosity of 702,5 for 7 g.l and R-squared (adjusted by g.l) = 99.6 %; therefore, the behavior of the production system must be regulated considering its actions depending on the factors. The data shows the fitted model with values of the variables and that are specified in their original units.

$$\mu = 702,5 + 395,75 X_1 + 61,625X_2 + 44,37 X_3 + 17,62X_1X_2 + 13,87 X_1X_3 - 18,25 X_2X_3 - 32,0 X_1X_2X_3$$

Eq.1.

The yield of the mucilage obtained is 1865 ml and 1683 ml at a concentration of 80g/2L and 160g/2L, respectively, depending on the degree of disintegration of the material, plant maturity stage and harvest season that deserve further investigation. Concentration, hydration time and stirring speed affect the yield of the mucilage. The soluble solids in the solution of the mucilage obtained was less than 2 °Brix and has a pH of 6.6.

Viscosity and amount of foam in the shampoo

Table 5. Viscosity and amount of foam

BLOQUE	Amount of Mucilage	Amount of Texapone	Viscosity	Amount of foam
	g.	g	mPa.s	ml.
1	-1	1	1350	192
1	1	-1	1720	146
1	1	1	2050	213
1	-1	-1	1020	144
2	-1	1	1330	190
2	1	-1	1760	151
2	1	1	2060	213
2	-1	-1	1215	145

Table 5 shows high values of viscosity and the amount of lather generated in the shampoo as the amount of Texapone and mucilage is incorporated at high levels. The viscosity of the shampoo using *Malachra alceifolia* Jacq mucilage as a thickener and substitute for sodium chloride is between 1020 and 2050 mPa.s. The formation of Amount of foam 145 to 213 ml., and is maintained for 6 minutes, enough time to cleanse the scalp and hair; Results of the experimentation in the shampoo using the mucilage as a thickener and substitute for sodium

chloride, tabulated for an R-squared (adjusted by g.l) at 99.11% and according to figure 5 of the response surface for viscosity, shows that the viscosity reaches higher readings as the level increases due to the effect of the amount of texapone and mucilage incorporated in the shampoo formulation. Consequently, the incidence of the viscosity in the final product depends on the quantity and thickening capacity of the incorporated mucilage.

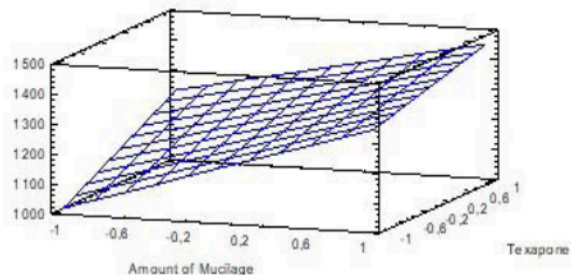


Figure 5. Response Surface for Viscosity (μ) of the shampoo

Those values are compatible to commercial products that are between 1000 and 9000 mPa.s and; the minimum viscosity of the shampoo³⁶. So, it does not flow into the eye, with a value of 1250 cP³⁷. The thickening capacity of the mucilage can replace the effect of the electrolyte (NaCl) on the viscosity of shampoos, since, in formulations that use 0.5% NaCl, the viscosity exceeds values of 200 cP and at 1% it could reach higher values. of 10,000 Cp³⁸, a shampoo typical of a non-Newtonian fluid³⁹. The leather formation in the shampoo is represented in figure 6.

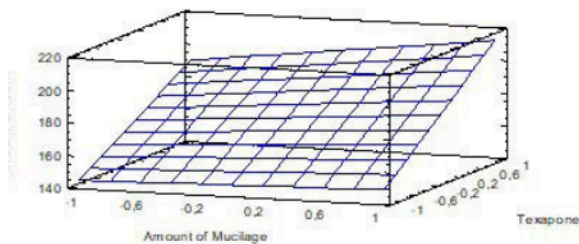


Figure 6: Response Surface for amount of foam

Diagram shows that it is influenced by the amount of high-level incorporated texapone and its ingredients, rather than the amount of mucilage hydrocolloid added in the shampoo formula. Shampoo formulas are produced with many natural substances that have a good viscosity, of leaf and flower extracts⁴⁰, but the use of mucilage as a thickener provides satisfactory results that allow new lines of research in Ecuador. It has a light green color, viscous appearance, homogeneous consistency, causes hair shine, softness, hydration capacity, clean appearance, pleasant aroma, ease of combing and characteristic shine are properties of the *Malachra alceifolia* Jacq. mucilage-based shampoo, they must be valued by the consumer. Expanding research to obtain the hydrocolloid with water from plant extracts with a high content of saponins will further qualify the

product and therefore its benefits. Herbal shampoo physical parameters such as appearance, viscosity, and pH are important in evaluating a shampoo³⁶. The shampoo obtained showed a pH between 6.8 and 7.0, a value similar to that of market products with a salt-free label. Hibiscus rosa-sinensis flower shampoo shows a pH equal to 6.5⁴¹. The mucilage is a natural moisturizer for the skin, antibiotic, anti-inflammatory and thickener substance commonly used in the traditional medicine of some Ecuadorian communities.

CONCLUSION

The complex molecular structure and abundant presence of mucilage in its natural state in the stems of *Malachra alceifolia* Jacq allows the hydrocolloid obtained to have a great viscosifying and moisturizing capacity with an almost neutral pH, qualities that affect the quality of the shampoo and are suitable for commercial products with friendly features to the environment; being promoted as a raw material for formulations of other personal hygiene products with an important projection of immediate application in the pharmaceutical and cosmetic industry.

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