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Nopal mucilage as hydration agent for quicklime; extraction methods

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Abstract: The main objective of this research is to select an extraction method of nopal mucilage to be used as a hydration agent at a suitable concentration for slaking quicklime in order to obtain lime putties with better rheological and mechanical properties. An experimental phase was performed where different extraction methods to obtain nopal mucilage were tested. This made it possible to classify them into two groups: methods that require water to get the mucilage, and methods that obtain the mucilage directly from the plant. Galacturonic acid present in mucilage composition seemed to be the main cause of the chemical interaction between lime and mucilage. As a result, mucilage that presented the largest amount of galacturonic acid according to acid base titration curves was chosen as the best one to work with. It was concluded that the amount of galacturonic acid present in mucilage is not influenced by rising temperature and constant stirring during its extraction process, nor the nopal species or cladodes dimension. Furthermore, it was observed that mucilage viscosity obtained through the different extraction methods has a strong impact on the slaking process behavior and in the consistency of the resulting putties.

Key words: slaked lime putty, nopal mucilage, galacturonic acid, quicklime slaking process, acid base titrations, extraction methods.

El mucílago de nopal como agente de hidratación de cal aérea; métodos de extracción

Resumen: Esta investigación tiene por objeto la elección de un método de extracción de mucílago de nopal para emplear a este último como agente hidratador de cal para producir cal apagada que confiera mejores propiedades reológicas y mecánicas a las pastas de cal apagada. En esta etapa se desarrolló una fase experimental que incluyó distintos métodos de extracción de mucílago de nopal siendo posible clasificarlos en dos grupos: los que emplean agua en su procedimiento y aquellos que obtienen el mucílago directamente de la planta sin adicionar otra sustancia. El ácido galacturónico parece ser la sustancia responsable de la interacción química entre el mucílago y la cal, por lo tanto, el mucilago que presentó la mayor cantidad de ácido galacturonico de acuerdo a las curvas de valoración ácido base, fue elegido. Pudo concluirse que la cantidad de ácido galacturónico presente en el mucílago de nopal no está influenciada por factores de elevación de la temperatura y agitación durante el método de extracción, ni por la especie y el tamaño de sus cladodios. Adicionalmente fue observado que la viscosidad del mucílago obtenido de los diferentes métodos de extracción tiene un impacto importante en el comportamiento del proceso de hidratación de cal y en la consistencia de las pastas de cal.

Palabras clave: Pasta de cal hidratada, mucílago de nopal, ácido galacturónico, procesos de apagado de cal, titraciones ácido base, métodos de extracción.

Introduction

Since ancient times, a number of different cultures that use lime in construction have known the benefits of using additives in the formulation of mortars in order to improve the properties of this compound (Barba and Villaseñor, 2013). An ancient production process from a north Mayan region consisted of slaking quicklime using water combined with mucilage from different kinds of tree bark. This practice indicated that mucilage could probably improve lime mortar properties much more effectively from the quicklime slaking process (Carrascosa and Lorenzo, 2012). Nopal mucilage is one of the most affordable additives for Mexico, in economic and geographical terms. This additive has exhibited favorable behavior when employed in the formulation of paste and restoration mixtures (Bedolla, 2009). It is made up of a wide range of polysaccharides and other substances, like vitamins and hormones, and it has a high viscosity (Nazareno and Padrón, 2011).

As a result, a hypothesis was drawn: if it has been demonstrated that nopal mucilage improves lime mortar properties like resistance, viscosity and workability when it is used during mixture formulation, then it could also improve lime mortar behavior if it is used in an appropriate dilution instead of using only water to hydrate or slake quicklime.

Therefore, the main objective of this stage of the research is to select a method to extract nopal mucilage that could be used as a hydration agent of quicklime to improve the properties of this binder as slaked lime putty from its production, and not only during mortar or mixture formulation (Pérez et al., in press).

Methodology

During a first phase of experimentation performed with students of the Faculty of Chemistry at UNAM (National Autonomous University of Mexico), nopal mucilage seemed to interact strongly with cations such as calcium Ca+, and it has been reported that the chemical substance responsible for this is galacturonic acid. Consequently, it was hypothesized that this substance was the main cause of the chemical interaction between lime and mucilage and that it produced the desired results in lime mortars (Carmona, 2015). Therefore, it was considered that the mucilage that presented the largest amount of galacturonic acid in its composition would be the one that would display a better behavior in the lime hydration phase to produce slaked lime putty with better properties of application and resistance. The ultimate aim of the research was to develop an extraction method that would produce a greater volume of mucilage and facilitate the dynamics of the restoration work as much as possible.

Extraction methods were reproduced as described in investigations by various authors, and some adjustments were made based on experience.

The extraction methods tested were classified into two groups: the ones that need water to obtain the mucilage (constant ratio followed: 1:2, nopal: water by weight) [Figure 1a, b, c], and the ones that obtain the mucilage directly from the plant without any other substance [Figure 2a, b, c] [Table 1]. The resulting product from each method is considered as a 100% solution of mucilage.

Figure 1.- 1a, 1b, 1c: An example of extraction methods that need water to obtain the mucilage. 1a: Cladodes are chopped into small pieces, 1b: Cladodes are poured into water in a proportion of 1:2 (nopal : water in weight) for about 24 hours, 1c: Nopal mucilage is sieved through different sized meshes. Pérez et al., "The hydration of lime using nopal mucilage to optimize hydrated lime mortars for the conservation of built heritage". MNCARS, Madrid, Spain, in press.

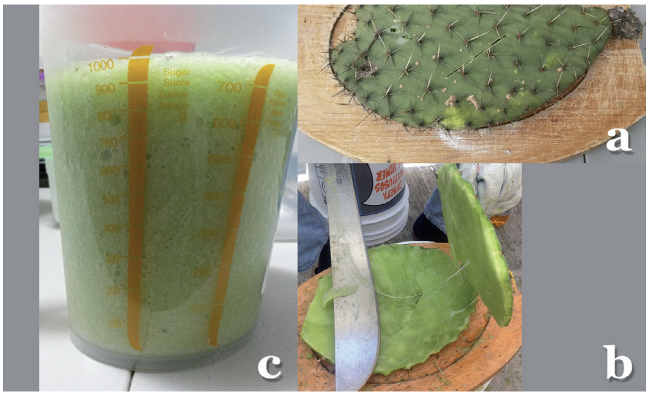

Figure 2.- 2a, 2b, 2c: An example of extraction methods that obtain nopal mucilage directly from the plant. 2a-2b: cladodes are cut halflenghtwise, 2c: Cladodes inner faces are scrapped in order to remove mucilage directly from the plant. Pérez et al., "The hydration of lime using nopal mucilage to optimize hydrated lime mortars for the conservation of built heritage". MNCARS, Madrid, Spain, in press.

Table 1.- Extraction methods of nopal mucilage. Pérez et al., "The hydration of lime using nopal mucilage to optimize hydrated lime mortars for the conservation of built heritage". MNCARS, Madrid, Spain, in press.

Methods of extraction of nopal mucilage								
Method No.	Species used	Elements removed	Cladodes cut	Proportion in weight nopal:water	Appliance used	Temperature (°C)	Stirring	Mesh apperture
Methods that use water in its procedure								
1	Tlaxcalancingo Puebla	Cutin, epidermis and spines	Chopped in pieces (2cm)	1:2	liquefied	70-80	occasional	2mm-1mm-12-15μm
2	Tlaxcalancingo Puebla	Cutin, epidermis and spines	Chopped in pieces (2cm)	1:2	liquefied	70-80	occasional	not meshed
3	Tlaxcalancingo Puebla	Cutin, epidermis and spines	Chopped in pieces (2cm)	1:2	/	/	/	2mm
4	Supermarket	Cutin, epidermis and spines	Chopped in pieces (2cm)	1:2	/	80	constantly for 30 minutes	/
5	Tetla Tlaxcala	Cutin, epidermis and spines	Chopped in pieces (2cm)	1:2	/	80	constantly for 30 minutes	/
Methods that acquire the mucilage directly from the cladode								
6	Tlaxcalancingo Puebla	Spines one by one	Cut half lenghtwise	/	/	/	/	/
7	Tecali Puebla	Spines one by one	Cut half lenghtwise	/	/	/	/	/
8	Tetla Tlaxcala	Spines one by one	Cut half lenghtwise	/	/	/	/	/
9	Tetla Tlaxcala	Spines one by one	Cut half lenghtwise	/	/	40-60	constantly for 30 minutes	/

The species that were tested with the different extraction methods included Opuntia ficus-indica cultivated species, indigenous to Tlaxcalancingo, Puebla, a wild species from Tecali, Puebla and Tetla, Tlaxcala, and finally species purchased in supermarkets in Puebla City.

In the first extraction methods tested, cladodes of a 40cm length x 25cm approximate width from Tlaxcalancingo, Puebla, were used. In this method the cutin, epidermis, and spines were removed using a knife. The cladodes were chopped into small pieces of about 2cm length, liquefied in a blender (Oster-BLSTMG-MR) for about 5 minutes, poured into water using a proportion of 1:2 (nopal:water in weight) and then the temperature of the mixture was increased to between 70°C and 80°C on a standard kitchen stove and measured using an industrial mercury thermometer (Alla France). A portion of the extract was passed through meshes of different sizes, 2mm, 1mm (method No.1), while another amount was left unsieved (method No. 2).

Method No. 3 was a variant of the previous two, where nopal from the same origin was chopped (cutin, epidermis and spines were previously removed), and left in water using a proportion of 1:2 in weight (nopal:water) for 24 hours at room temperature. This time, its extract was passed through a 2mm mesh because, in the previous method, a great amount of solution was held in the 1mm mesh.

Extraction method No. 4 included some cladodes obtained in a supermarket that were chopped into pieces of 2cm, poured into water in a proportion of 1:2 (nopal:water), and exposed to a temperature of 80°C on a standard kitchen stove while being stirred constantly by hand with a plastic spoon for 30 minutes.

Extraction method No. 5 was developed as a variant of extraction method No. 4 and involved nopal cladodes native to Tetla, Tlaxcala, of approximately 65cm length x 30cm width, since it had been noted that a larger amount of mucilage in liters was obtained from each cladode. The extract was subjected to a temperature of 80°C on a standard kitchen stove and was stirred constantly by hand with a plastic spoon over a period of 30 minutes.

A sixth extraction method was developed using Tlaxcalancingo cladodes. This method reproduced a traditional technique of Otomi ethnicity (this method was obtained by direct observation). It involved cutting the cladode in half lengthwise and scraping the inner faces using a knife in order to remove the mucilage directly from the plant without using water as a solvent. This extraction technique was replicated using native cladodes from Tecali, Puebla, (method No. 7) and Tetla, Tlaxcala, (method No. 8).

Method No. 9 arose as a variant of extraction method No. 8, where the product obtained with the original technique (method No. 6) was stirred constantly for 30 minutes using a plastic spoon and heated on a kitchen stove to 40 - 60 °C. An industrial mercury thermometer was used to measure the temperature (Alla France).

Subsequently, acid-base titrations were performed to 25 mL of extracts obtained from each method with gradual additions of 0.25 to 0.50 ml of sodium hydroxide 2 x 10-2 M and the support of a pH meter METROHM 620 for pH measurements reflecting the results in titration curves that led to partial conclusions about the presence of galacturonic acid. The greater the amount of sodium hydroxide needed to produce a significant increase in pH to basic values in each extract, the greater the amount of galacturonic acid it had.

Results & discussion

All the results were analyzed, and the two methods that displayed the greatest amount of galacturonic acid and the largest quantity of mucilage from each group of extraction methods were selected in order to create a graph and explain the criteria used to choose which one to work with. As shown in the graph [Figure 3a] from the group of extraction methods that use water in its procedure, No.1 and No. 4 reported the greatest amount of galacturonic acid, while No.6 and No.8 were selected from the group of methods that obtain the mucilage directly from the plant.

From the group of extraction methods that use water in its procedure, method No. 4, which involved an increase in temperature and constant stirring, reflected the highest amount of galacturonic acid in its composition. During titration process, the starting pH was 3.85 and relatively increased in values of 0.05 to basic standards every 0.25mL of sodium hydroxide added. A considerable increase of 0.30 in pH to basic values occurred after a concentration of 8.5mL of Sodium hydroxide had been added to the mucilage. Method No 1., which started with a pH of 4.2 and reported a slight increase in values of 0.05 every 0.25mL of sodium hydroxide added, until a significant increase of 0.10 in pH was reported when 8.00mL of sodium hydroxide was added to mucilage.

From extraction methods that obtain the mucilage directly from the plant, the mucilage obtained by method No. 6, which used cladodes from Tlaxcalancingo, Puebla, was shown to contain the greatest amount of galacturonic acid [Figure 3a]. The starting pH was 4.75 and there were small increases of 0.05 in pH every 0.25mL of sodium hydroxide added, but a more significant surge of 0.20 in pH to basic values took place when 16mL of sodium hydroxide was added. Method No. 8 seemed to contain less galacturonic acid than No. 6. It reported an initial pH of 4.85 and increases of 0.05 in pH every 0.25mL of sodium hydroxide added and a more significant increase of 0.20 at 14.25mL of sodium hydroxide present in the mucilage.

As explained previously, the greater the amount of sodium hydroxide needed to produce a significant increase in pH to basic values in each extract, the more galacturonic acid the mucilage contained. As a result, extraction method No. 4 from the group of extraction methods that use water in its procedure and method No. 6 from the group of extraction methods that obtain the mucilage directly from the plant were chosen as the ones with the greatest amount of galacturonic acid in its mucilage.

The same graph [Figure 3b] also shows the volume of the mucilage produced in each method. Again, the two methods from each group of extraction methods that presented the largest amount of mucilaginous product were selected. As shown in the graph [Figure 3b], method No. 5 produced the greatest quantity of mucilage from the group of extraction methods that use water in its procedure, since each cladode indigenous to Tetla, Tlaxcala, of a 2.7kg average weight plus 5.4kg of water (following a constant ratio 1:2 nopal: water in weight) produced 6L of mucilage. In second place from the same group of extraction methods, No. 1 produced 4 L of mucilage from 1.76kg of nopal plus 3.52kg of water.

From the group of extraction methods that obtain the mucilage directly from the plant, methods No. 8 and No. 9 seemed to be the ones that produced the greatest amount (L) of this substance since 1.5L was obtained from each cladode of about 2.7kg from Tetla, Tlaxcala.

Not only is it worthy to choose an extraction method that produces mucilage with the greatest amount of galacturonic acid in its composition, but also a method that produces an abundant amount of the mucilaginous substance in liters in order to best facilitate the restoration

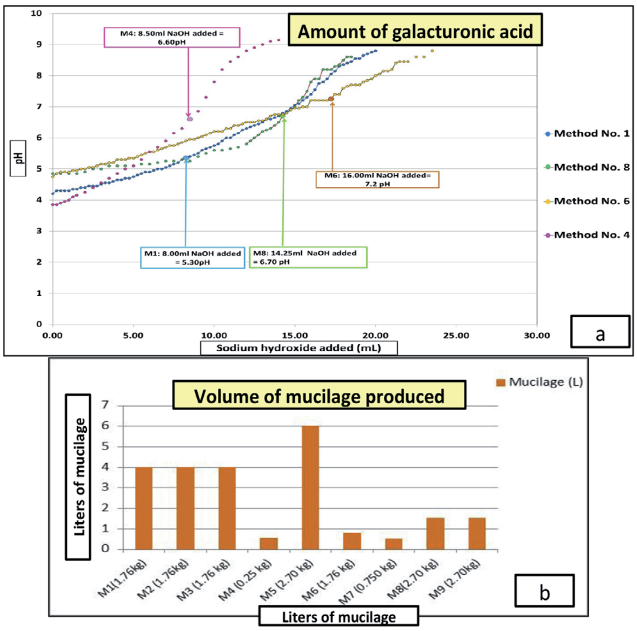

Figure 3.- 3a-3b: Amount of galacturonic acid in mucilage and volume of mucilage produced from the different extraction methods. 3a: The two methods that displayed the greatest amount of galacturonic acid from each group of extraction methods tested. 3b: The two methods that displayed the largest quantity of mucilage obtained from each group of extraction methods tested. Pérez et al. "The hydration of lime using nopal mucilage to optimize hydrated lime mortars for the conservation of built heritage". MNCARS, Madrid, Spain, in press.

activities at a construction site. Both factors were considered in order to select the best extraction method, but priority was given to the content of galacturonic acid.

After having analyzed the results, it was concluded that method No. 8, which belongs to the group of methods that obtain the mucilage directly from the plant, reported an abundant amount of galacturonic acid in its structure and produced a worthy volume of mucilage, so it was selected as the best to work with as a hydration agent for quicklime.

Slaking quicklime using mucilage

A first experimental phase was also developed for slaking quicklime (CaO) using the mucilage chosen (the one from method No.8).

In the traditional slaking quicklime process using only water as hydration agent, a sudden violent reaction is produced in which a sharp increase in temperature to between 90°C and 100°C takes place and the resulting putty shows a homogenous consistency.

In this experimental stage using mucilage solution as a hydration agent instead of only water, the temperature was measured with an industrial thermometer and consistency was evaluated by observation in order to compare both processes (the original one using only water as hydration agent and this investigation's suggested process: using mucilage solutions to slake quicklime) and their resulting lime putties.

To develop the experiment of the investigation plan, it was decided to begin with mucilage solutions in three different concentrations in water, 100%, 60% and 20%, as a first proposal using the selected mucilage.

To begin with the lime hydration or slaking process, a ratio of 1:3 was chosen (quicklime: mucilage solution in weight). The selected ratio was the one that proved to be appropriate according to experience because, as the authors mentioned, the amounts of water needed in theory to hydrate quicklime and to obtain slaked lime putties do not work in practice, and there is even the risk of drowning (Boynton, 1980; Bedolla, 2010).

The starting temperature of the quicklime slaking process did not go above 40°C in any of the three cases and the violent reaction took between 10 and 15 minutes to start. At the beginning, the process produced an agglutinate, yellowish sub product, which was stirred for 1 hour. According to Robert Boynton's investigation, the temperature reported might reflect a decrease in the surface area of the hydrated lime crystals and therefore a significant decrease in the rheological properties of the resulting slaked lime. In this time, the stirring of the sub product resulted in another high-viscosity sub product, of heterogeneous appearance which could not be handled as integral lime putty to produce a mortar. Once the process finished and the final product cooled, this was left to rest on a 5-cm-thick mirror solution of nopal mucilage in water in order to protect the putties from the process of carbonation which takes place with the carbon dioxide in the air. Two months later, the final product was inspected and it was confirmed that it kept the same characteristics, like consistency and lumpy heterogeneous appearance, without improving its rheological properties after this time. From this procedure, it can be concluded that such a high viscosity of nopal mucilage as obtained though this extraction method did not allow the reaction to take place in an optimum way.

Consequently, the quicklime slaking process using mucilage was reproduced again but this time using mucilage extracted by method No.5 which uses water in its procedure and includes a rise in temperature and constant stirring as variables.

In the second experiment, the lime slaking process was carried out in a very similar way to the previous one, mucilage solutions in three different concentrations in water 100%, 60% and 20% were used as hydration agents with a proportion of 1:3, quicklime to hydration agent. This time the quicklime slaking process was also undertaken using water with no additions as a control test.

The exothermic reaction of the process occurred immediately. The whole procedure took approximately 2 hours. The 5-cm-thick nopal mucilage mirror was used again for the resulting putty from each process (20%, 60% and 100% mucilage solutions) and a water mirror of the same thickness for the slaking lime process, which used only water as hydration agent to prevent carbonation. The most violent reaction and the highest rise in temperature (105°C) during the slaking process was reported by the process that used only water as hydration agent, followed by the one that took place at 20%, mucilage solution, then 60% and finally 100%. All of them reached a temperature of between 90°C and 100°C. In this experimental phase, touch and sight reported the best consistency in the resulting putty in the nopal mucilage at 20%. In a subsequent phase, consistency will be evaluated using a Bostwick ZXCON consistometer.

Conclusions

Extraction methods which include water in the process offer the advantage of producing a larger amount of mucilaginous substance (as it is shown in the graph for method No. 5 the one that produced the largest volume of mucilage), but demand a period of at least 24 hours and report a lower amount of galacturonic acid than those that do not use water in its procedure. In contrast, extraction methods that obtain the mucilage directly from the cladode can reduce the production time and offer a higher content of galacturonic acid in its composition. However, the volume obtained is lower compared to the first group.
The rise in temperature and constant stirring during extraction processes are not related to the amount of galacturonic acid contained in the mucilage acquired since method No. 6, which does not include a rise in temperature and constant stirring, reported the greatest amount of this chemical substance from the second group of extraction methods.

The species that produced the largest amount of mucilage were the wild cladodes from Tetla, Tlaxcala, and Tecali, Puebla, in both groups of extraction methods.

Mucilage viscosity obtained through the extraction methods which do not use water in the process is so high that the reaction is very slow when it is used as a hydration agent of quicklime. This results in low quality slaked lime putties of heterogeneous consistency that are unsuitable for use as a binder in lime mortars. On the other hand, mucilage that includes water in its extraction process allowed an exothermic reaction to occur immediately during slaking lime process, reached a temperature of 100°C, and produced lime putties with better a consistency than the previous and will be used as binder for lime mortars in further experimental phases.

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