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Full Length Research Paper

Thermal effects on granules and direct determination of swelling capacity of starch from a cassava cultivar (Attiéké Mossi 1) cultivated in Côte D'Ivoire

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Food security assurance is a vital challenge that is still facing Africa today. Thus, numerous research topics have been oriented toward this agronomic aspect in order to increase food production. This is one of the reasons researches on crops were disregarded. This lack of scientific results led us to investigate the evolution of the granules size of cassava starch, according to the temperature and heating duration. It permitted the division of the ranges of temperature into 2 groups. The first, which consisted of 60, 65 and 70°C, was not able to induce starch gelatinization during the 30 min of heating; the second group, which consisted of 75, 80, 85 and 90°C, provoked the complete starch gelatinization before the end of the heating. This survey also confirmed that the small granules (diameter ≤ 10 µm) made a more elevated resistance than the thickest ones. Moreover, it confirmed that during the heating, the starch granules swelled to reach an optimum level of absorption before reaching their solubility. This observation led us to propose another determination method of swelling capacity than that of the Leach's method. The so-called method, not only considered the mean size of granules at the optimum period of absorption, but also took into account total water absorbed by granules, since it was valued according to their size. The application of this method produced the swelling of the granules which was extensively elevated than that of Leach. At 90°C for example, the proposed method induced a swelling (73.09 g of water/g) raised 4 times more than the Leach's method.

Key words: Thermal, effects, swelling, capacity, cassava, starch, Côte d'Ivoire.

INTRODUCTION

In Africa, the worry to guarantee food security oriented almost all the researches towards agronomic aspect of starchy plants, in order to improve their productivities (FAO, 1998). So the investigations aiming to add more worth to the crops were forgotten. This is how studies on starch, the major constituent of the so called plants, which is very important because of its numerous implications, have been disregarded. For instance, only 12% of scientific publications on this derivative product concern the African ones, in which 5.8% is for cassava starch (FAO, 1998).

In spite of this, area that concerns starch is still experiencing rapid growth. It is extensively used in food industry, particularly growth in the domain of cooked dishes; also more and more in paper industry, in medicine industry and in the chemistry of plastics. In this last

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domain starch can substitute oil derivatives with advantage to making them biodegradable (Massaux et al., 2006). In addition, starch proved to be more effective and availably cheaper among several other natural polymers (Mali et al., 2005; Mali et al., 2004; Rindlav-Westling et al., 1998; Tomka, 1991).

Starch usage in these ways is possible only if many researches are done on its physical and chemical properties, such as its viscosity, its gelatinization, and its shearing rate. These hydrothermal properties are themselves known as closely dependent on the morphology and the individual and collective hydrofunctional behavior of starch granules (Massaux et al., 2006). They also depend on the amylose/amylopectin ratio of starch (Rindlav-Westling et al., 1998). These same intrinsic properties are known as affectable by the conditions and preparation methods (Flores et al., 2007; Myllarinen et al., 2002; Rindlav et al., 1997; Rindlav-Westling et al., 1998; Rindlav-Westling et al., 2002; vanSoest et al., 1996a; vanSoest et al., 1996b). For instance, the strength and the viscosity of a starch gel depend on the temperature and its cooking time (Doublier et al., 1987; Flores et al., 2007).

It is also reported that starch rich in small granules is more resistant to the external influences, therefore, difficult to transform. This same starch, because of its elevated lipids content, is reported to be more resistant to swelling than those rich in thick granules (Massaux et al., 2006).

This paper reports on the swelling of two populations of granules in the starch of a cassava cultivar (attiéké Mossi 1), a starch poor in lipid (Sidibé et al., 2007). This work would also permit avoidance of the loss of energy that occurs during prolonged heating, a factor that produces, unfortunately, the degradation of the starch gel (Doublier et al., 1987).

MATERIALS AND METHODS

Material

The cassava roots that constituted the biological material were harvested from CNRA experimental plot (Adiopodoumé, Côte d'Ivoire).

Starch extraction

The starch was extracted through the humid way according to Banks and Greenwood's method (1975), modified by Amani et al., (1993).

Morphological, granules size and statistical distribution analysis

A pinch of starch flour was put on a slide. The granules were stained with a drop of cedar oil for a microscopic clarity. The observation was made through a system that consisted of a light microscope (CETI), equipped with a TK-C721EG recording camera (JVC instrument) connected to a surveillance monitor TM-1700PN (JVC instrument) and to a computer via an acquirement card. The microscope was piloted by a computer through an adapted software (KAPPA) that was able to capture images and to take granules size. Several images were taken for analysis.

Starch granule sizes evolution, during heating

The heating temperature range went from 60 to 90°C with a regular spacing of 5°C (60, 65…. 90°C). For each temperature, the starch suspension (4%, w/w) previously prepared in the distilled water and staked in a 150 cc beaker, was heated during 30 min in a viscosimetric water bath MD 18 V equipped with a thermostat and digital display (JULABO instrument). For each temperature a new suspension was constituted and the operation was performed in triplicate. Microscopic observations were made for each temperature every 5 min. And at least 100 images were captured. On these images, the sizes of 1000 granules were taken. The behavior of all the granules present at this instant in the heating environment was appreciated by the determination of the mean diameter and the standard deviation (Larrigue et al., 2008) of those present in observed samples. Indeed, all the variations of standard deviation in the treatment environment compared to the native starch, express a difference in granules population resistance, because if the granules resisted with the same ardor, there would have a homogeneous swelling relatively proportional to their initial size. If the variation of the standard deviation was a growth, it suggested that the thick granules resisted less to the thermal treatment than the small ones, because if the small ones were more vulnerable, the heating would swell them to reach or to bring closer to the size of the thicker ones. That would decrease the distribution extent (standard deviation). So a decrease of the standard deviation expresses a more increased resistance for the thick granules than the small ones.

Determination of swelling capacity according to Leach's method (1959)

A starch suspension (4% w/w) was heated. After centrifugation the sediment was weighed, dried at 120°C during 1 h 30 min and weighed once more. The sediment water content corresponding to the swelling capacity (g of water/g of starch) was determined by the following formula:

$$\frac{P1 - P2}{P}$$

Where P1 is the weight of the sediment, P2 is the weight of the dried sediment and P that of the starch used to achieve the suspension.

Direct determination of the swelling capacity

The appreciation of the swelling capacity should be made rightly when most of the granules acquired their maximum size. The study made above showed that starch stopped swelling at a precise moment and started solubilising. In addition, since the studied starch in majority consisted of thick granules, it adopted the behavior of the last one. Therefore the precocious swelling of the thick granules announced by Massaux et al. (2006) should be confounded to the swelling of the whole starch, and gave us the opportunity to determine the swelling capacity at a precise time. Certainly the determination of the swelling capacity at any heating duration led to an underestimation of this one. We propose to study the swelling capacity in this work, according to granules size because they are almost spherical. This method will have the merit to consider only granules water contents and not water agglomerated on the starch gel surface; and also the interstitial ones, as in the method of Leach (1959).

To perform the method, it was necessary to know the native starch granules mean volume (Vmg). It was expressed in accordance with the one of a sphere by the following formula:

$$\text{Vmg} = \frac{4}{3}\pi\left(\frac{d}{2}\right)^3 \quad (1)$$

Where d is the mean diameter of the native starch. The determination of the specific volume of the native starch (ρ) was also necessary. It was determined by a helium pycnometer AccuPyc 1330 (Micromeritics instrument). Since the final result should be expressed in "g of water/g of starch", granules number per gram of native starch ($N_{gr/g}$) was determined according to the following equation:

$$N_{gr/g} = \frac{1}{\rho\,\text{Vmg}} \quad (2)$$

$$(1)\ and\ (2) \Rightarrow N_{gr/g} = \frac{6}{\rho\pi}\left(\frac{1}{d}\right)^3 \quad (3)$$

Indeed, the swelling capacity Q (g of water/g of starch) is

$$Q = N_{gr/g} \times V_{Mg} \quad (4)$$

Where V_{Mg} is the swelling capacity of a swollen granule. According to (1),

$$V_{Mg} = \frac{4}{3}\pi\left(\frac{D-d}{2}\right)^3 \quad (5)$$

Where D is the optimum mean diameter acquired by granules during the heating.

$$(3),(4)\ and\ (5) \Rightarrow Q = \frac{1}{\rho}\left(\frac{D-d}{d}\right)^3$$

Expressed in "g of water/g of starch" if ρ is in "g/cm^3 or ml.

Statistical analysis of results

Variances were compared according to the ANOVA method through STATISTICA software.

RESULTS AND DISCUSSION

Starch extraction

The starch extraction according to the applied method has succeeded in making flour, with its hue that was extremely white, moisture content, 13%, in accordance with the norm of Thailand trade ministry (1993). Its granules were almost spherical (Photo 1). The extracted quantity represented 8.3% of the total mass of the cassava used. The starch content was lower than the one indicated by Maneepun (2000) (20%), and could be due to several reasons: probably the weakness of the starch content of the studied cassava, the loss in starch during the four decantations or the use of an unsuitable grinder, which was a screw one, rather than a hammer grinder which usually frees more starches.

The statistical distribution of the starch granules showed that granules sizes vary from 4.36 to 24.4 µm with the average at 11.32 µm (Figure 1). The studied starch consisted of 23.8% of small granules and 76.2% of thick granules on the basis of the classification of Massaux et al. (2006). Then it could be used in chemistry of plastics, because starches having diameters in this interval are usable in this sense (Massaux et al., 2006). The studied starch granules had appreciably the same size as those of the cultivar Bonoua 2 (2-25 µm) (Kouakou, 2004). On the other hand, they presented smaller size than those of the cultivars Bonoua akpessé (5-45 µm) and 524 (1-41 µm) (Kouakou, 2004). The granules of this starch were distinctly bigger than those of rice (3-8 µm), of smooth pea (5-10 µm) (Duprat et al., 1980) and distinctly smaller than those of yam (Amani et al., 1993).

Starch granule sizes evolution, during the heating

The behavior of granules, during the heating led to the division of the temperatures range into 2 categories. The first, which consisted of 60, 65 and 70°C, was not able to induce starch gelatinization during the 30 min of heating; the second, which consisted of 75, 80, 85 and 90°C, inflicted a thermal aggression inducing the complete gelatinization of starch before the first 25 min of heating. A similar observation was made by Bhavesh and Koushik (2006).

At 60°C, the graph showed the granules with their sizes increasing continually during the heating. Their swelling was petty. This light gain in size was illustrated by the pace of outcrop as shown on the graph (Figure 2). It meant that at this temperature, time was not sufficient to induce the swelling necessary to the bursting of granules, or this temperature was not aggressive enough to make granules acquire the maximum size resulting in the bursting. The microscopic observation of the heating environment confirmed this analysis (Photo 1A, B, C, D, E and F). At 30 min, granules grew in average 24.03%.

At 65°C, the average size evolution (Figure 2) showed globally, three phases. The first began at 0 to end at 10 min and allowed granules only to reach a mean size of 13.06 µm. This small variation was explained by the resistance that the granules opposed to the thermal shock. However, the second phase was represented by a

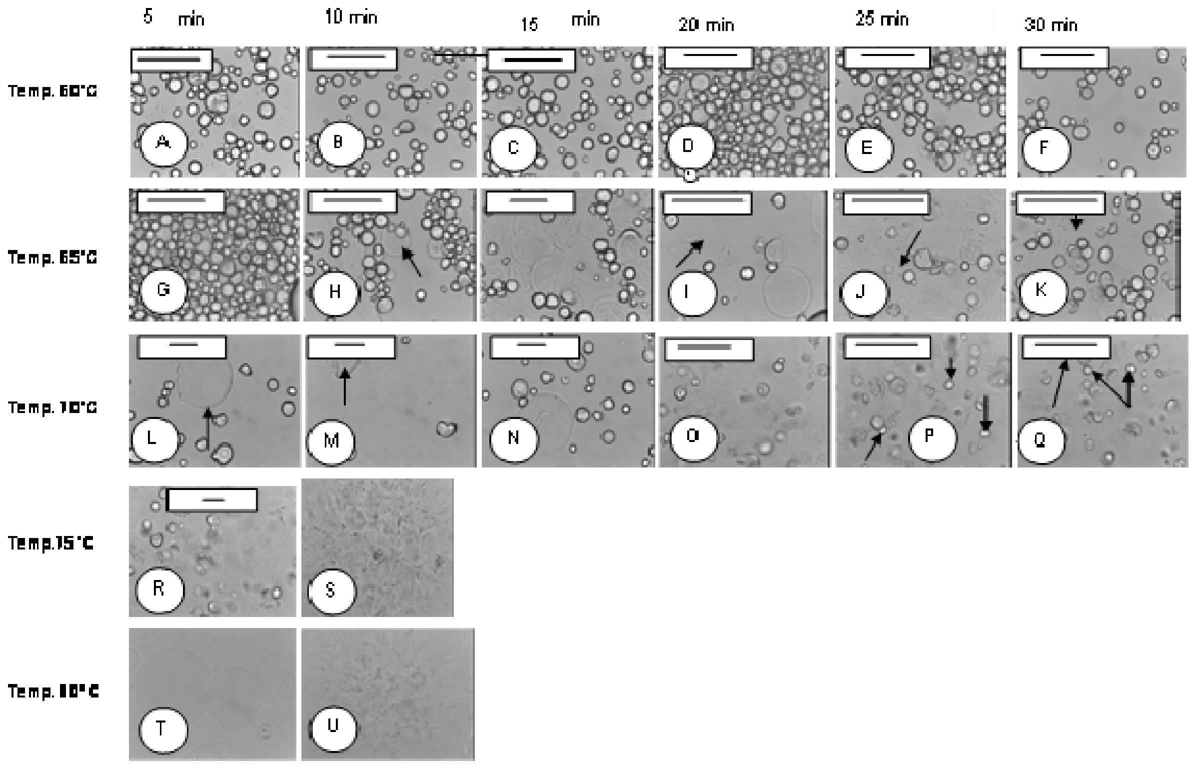

Photo 1. Evolution of granules sizes according to temperature and heating duration; bar in each micrograph is 50 µm in length.

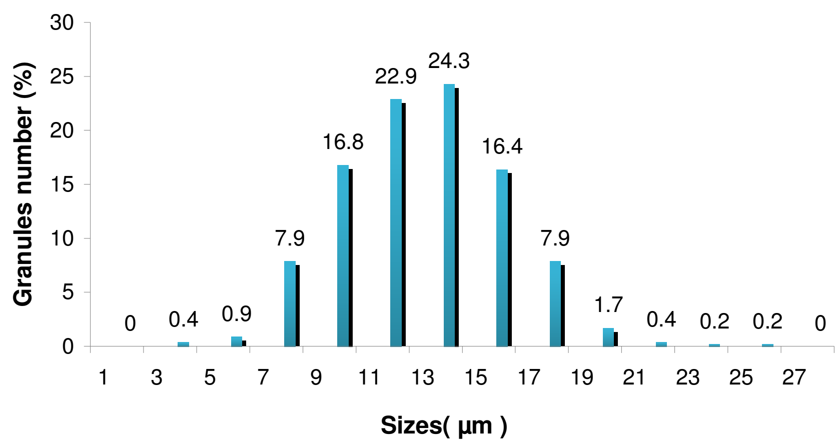

Figure 1. Native starch granules size distribution.

strong slope between 10 and 15 min, where granules reached their maximum size at this temperature, with a gain in size of 63.6%, far below the observation made by Tetchi et al. (2007) (210%) on another cassava variety. This brutal growth due to a massive absorption of water announced the end of the resistance of many granules. The last phase presented a decrease of sizes in the treatment environment. This decrease was due to the bursting of granules previously swollen. So it only remained what had resisted. The return nearly to the average size of departure attested that small granules resisted and therefore, they only stayed after the heating time. The small granules in majority enlarged to reach the mean size of the native starch. This analysis was observable on Photos 1I, J and K where we could observe a starch gel sprinkled on small granules. At this temperature the

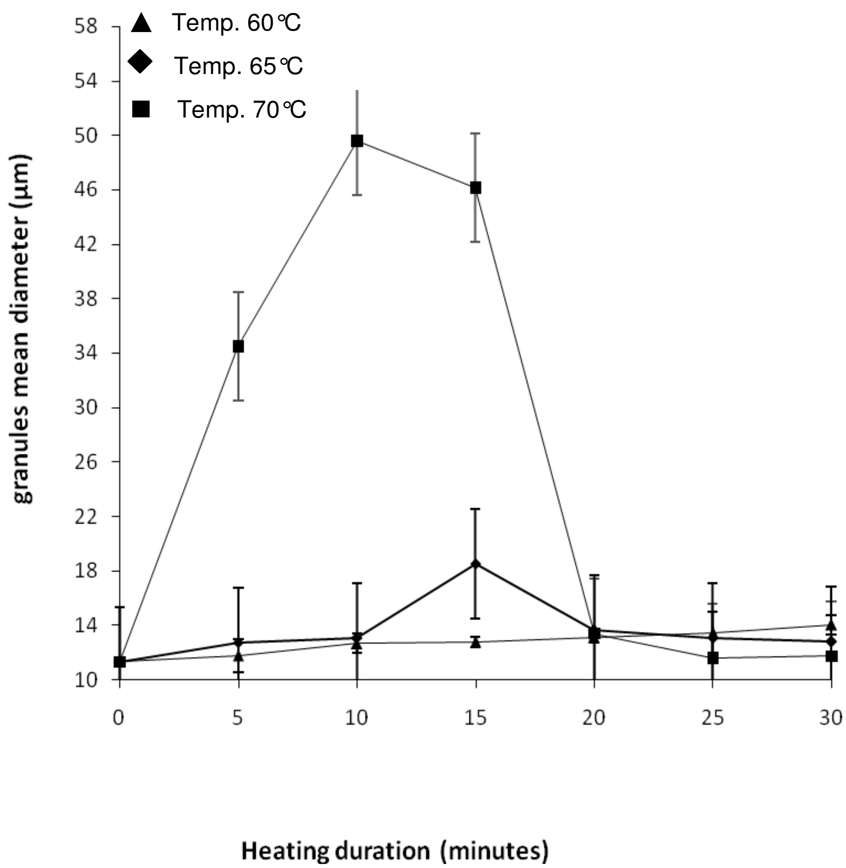

Figure 2. Evolution of the granules mean diameter according to heating duration. The ordinate at the abscissa 0 is the mean size of native starch.

heating time used in this study was not therefore sufficient to induce the complete gelatinization. So, this observation confirmed the small granules resistance (Massaux et al., 2006). But the quasi-absence of lipids in this starch certainly showed that this resistance was not solely due to the surface lipids. Other factors must be implied in this behavior.

At 70°C, the average size evolution (Figure 2) presented globally two phases. There was not a period of resistance, the massive absorption of water started simultaneously with the heating and induced a gain of size of 332.16% in 10 min. Arrows indicate granules in turgescence on the Photos 1L and M. The transformation into gel started after this period, therefore the decrease of size average in the treatment environment. Observations and analyses are similar to those described above. The same observations are made by Sabrina et al. (2008) about another cultivar of cassava. Compared to the works of Bhavesh and Koushik (2006), this starch swells more than the wheat one. Its use as thickener shall be then more beneficial and advantageous than the one of wheat.

The second set of temperature induced the complete gelatinization during the heating.

At 75°C, the complete gelatinization was observed at 25 min, during which the mean size was 53.80 µm, slightly over what was observed by Tetchi et al. (2007) (46.4 µm). Then all supplementary heating beyond 25 min should correspond to a wasting of energy and a destruction of gel for which starches are generally coveted. This gelatinization was precocious than the one observed by Sabrina et al. (2008). So, the industrial use of this starch will permit an economy of energy in relation to the one of Sabrina et al. (2008).

The temperatures 80, 85 and 90°C induced the complete gelatinization respectively at 15, 5 and 3 min. Beyond these times we could observe only ghosts (Atkin et al., 1998) in accordance with the observation made by Sabrina et al. (2008) at 95°C. Before the gelatinization the mean sizes of granules respectively reached 55.27, 62.03 and 65.65 µm. At these temperatures the swelling and the bursting of granules occurred in few minutes. No granule posed a resistance against the thermal shock.

The increase of standard deviations during the heating confirmed that the small granules resisted more than the thick ones. The decrease after the growth expressed the bursting of the thick granules previously swollen. Thus, the extent of the distribution reduced and approached the

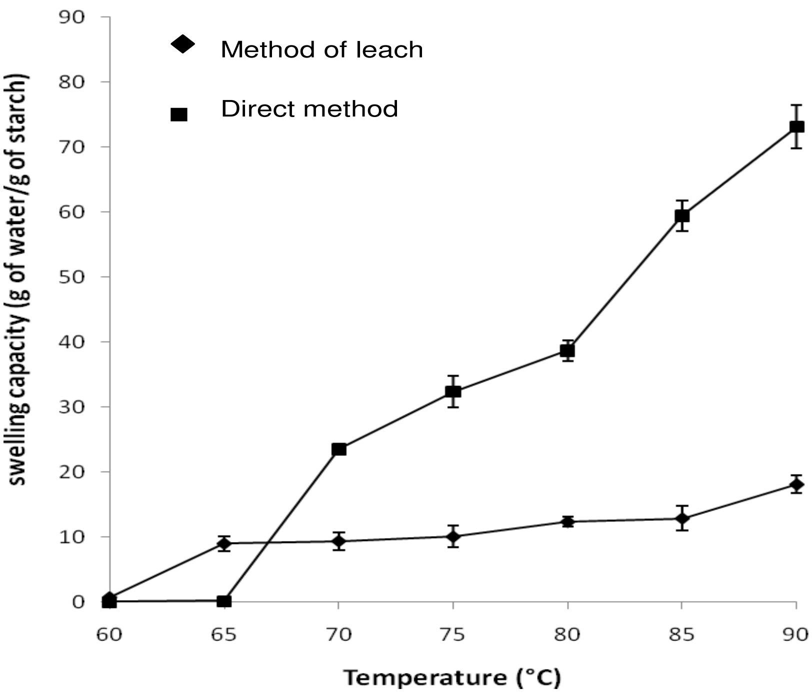

Figure 3. Evolution of swelling capacities according to the temperature.

native starch one. This confirmed the presence of the small granules only in this environment at this instant.

Swelling capacity studied by the two proposed methods

The specific volume of the starch determined by the helium pycnometer was 1,514 ± 0.002 g/cm^3. This value, in accordance with the swelling formula described above permitted the development of the graph of Figure 3. The diagram of Leach's method presented a regular growth from 0.008 g of water/g of starch at 60°C to reach 18.77 g of water/g of starch at 90°C. Whereas the direct method expressed an insignificant and negligible swelling from 60 to 65°C, in conformity with the observations made by Tetchi et al. (2007). From that temperature, the graph presented a jump reaching 73.09 g of water/g of starch at 90°C, 4 times more than the one observed with Leach's method (1959) at the same temperature. The swelling at 60 and 65°C were more raised for Leach's method (1959) than the direct method one by the fact that the former considered the granules surface waters, which was not the case in the second method which considered only granules inside water. Since at this temperature water absorption was petty, the swelling would be so. But from 70°C, since Leach's method (1959) was not applied at the optimum periods of granules absorption at the chosen temperatures, the direct method took over; despite the fact that it did not take into account interstitial and agglomerate water, because it was made at the right periods. Therefore, it expressed the real swollen capacity of starches, having granules that were spherical. The microscopic observation of the heating environment as descrybed above showed that granules swelled to reach a maximum size before exploding and dissolving before the 30 min, for most of the chosen temperatures. Therefore, the real appreciation of the swelling capacity must not be made at any instant but rightly at the period of optimum water absorption. Otherwise, it would be underestimated. Our result is the contrary of what other authors observed (Larrigue et al., 2008; Nayouf et al., 2003; Atkin et al., 1998) because they made the measurement at any time. But if the swelling capacity according to leach's method was appreciated at the period of optimum water absorption, it was always slightly over the direct one (Figure 4).

The statistical treatment of data presented a meaningful difference between results of the two methods at the risk of 5%.

Conclusion

This study confirms that the small granules have more elevated resistance compared to the thickest. But since this starch is very poor in lipid we can suggest that this resistance, in addition to factors expressed by Massaux is caused by other reasons. It could be about the restricttion of the heat attack surface compared to the thick granules. Further studies on starches provided on lipids

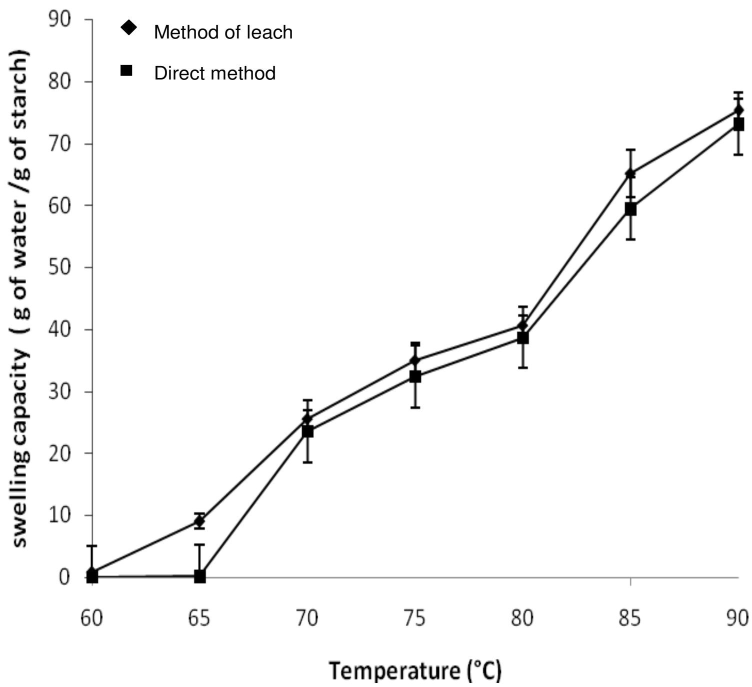

Figure 4. Evolution of swelling capacities according to the temperature.

could clarify factors involved in the resistance of small granules.

The swelling capacity determined according to the proposed method in this paper is more raised than the one expressed according to Leach's method. This confirms our anxiety regarding the Leach's method.

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