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EXTRACTION, CHARACTERIZATION AND EFFECTS OF COMPRESSIVE FORCE ON A NATURAL BIOPOLYMER

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ABSTRACT
The objective of this work is to study the modifications of the physical and structural treatments on the properties of taro starch (*Xanthosoma sagittifolium*) a species cultivated in Côte d'Ivoire and used in human nutrition compared to a standard corn starch. The influence of treatments in a context where sustainable development appears to be a major priority is part of a broad theme of valorization of agro-resources and the development of transformation processes devoted to renewable carbon resources. The characterization of the modifications of the physical properties of starch, generated by the treatments, was carried out with the aim of linking the differences of the native starches with the behavior of certain starches already used in the pharmaceutical field as excipients (analysis of images, hardness, densification, specific surface area and porosity). The physical treatments applied to the starches led to more or less significant modifications of their structures. The results of this study are even favorable to the use of this starch as a diluent in pharmaceutical forms.

KEYWORDS: Extraction, characterization, compression, *Xanthosoma sagittifolium.*

INTRODUCTION
Taros are among the world's leading starchy root crops grown in the world. These plants grow in the tropical and subtropical humid regions of the Ivory Coast where they are of cultural importance. However, Côte d'Ivoire, like some African countries such as Burundi, Uganda, Rwanda, use almost all the volume of this production available internally to feed people as a subsistence crop par excellence. This situation has led to a greater presence of potato products and cereals on the international market compared to those of African countries (Amani et al., 1993).

Despite their obvious economic role and a high production of these starches observed in these countries, very little work is devoted to them scientifically for the production of starch in an industrial way (Yobouet, 1999). The valorization of these local products turns out to be an important axis of economic and intellectual development. The use of these starches as pharmaceutical excipients constitutes a promising route.

The purpose of this study is to explore their possible use as an excipient (diluent) in the direct compression tablet formulation after characterization of these raw materials (electron microscopy image analysis, hardness, porosity).

1. MATERIAL AND METHODS
1.1. Material
It is composed of amylopectin No. A7780 lot 1221 H0265 provided by SIGMA laboratories, corn starch No. AC 8608 lot 9005-25-8 of ANACHEMIA chemicals limited, two varieties of the genus *Xanthosoma* (taro with red and white flesh), all grown in Côte d'Ivoire and harvested six months after planting. An EDWARDS auto 306 evaporator, a JSM 840 scanning electron microscope, a SPEAC England hydraulic press, an ERWEKA Pharmatest PTB 301 hardness tester (HAMBURG-Germany) and a COULTER SA 3100 gas prostheter, a typical PHILIPS mixer CUCIRA for the grinding of tubers, sieves of standard AFNOR PROLABO type whose meshes are respectively 0.315 µm, 0.2 µm, 0.125 µm in diameter

and a balance of SARTORIUS precision type 2462 with digital display with an accuracy of 0. 01 g.

1.2. Extraction of starches

The starches are extracted by the wet method according to the method described by BANKS and GREENWOOD in 1975 and modified by AMANI (**Amani and al., 1993**). The tubers are peeled, washed thoroughly, and sliced and soaked in sodium bisulfite solution (0.1%, w / v) for 24 hours to prevent oxidation of the starch. The slices are crushed in a PHILIPS CUCIRA mixer and allowed to stay in water for one day. The extractions are made with water at basic pH (9-10). The milk obtained is then taken up in a solution of 4% Nacl, P / V in order to separate the proteins from the starch and then passed through a series of sieves. The starch milk obtained undergoes alternating decantation washing (4 times at least). The supernatant is poured and the deposit obtained is spread on aluminum foil then dried in a ventilated oven (MEMMERT 1000 WATTS) at 45° C. for 48 hours. After drying, the dry product is pulverized in a porcelain mortar with a pestle to obtain the starch powder. This powder is weighed and packaged in a sealed jar.

1.3. Scanning electron microscope (SEM) image analysis

The preliminary study of the granulometry and morphology of starch grains was made by scanning electron microscope; the analysis of the tablets both on the axial surface and on the radial plane (tablet section) was also performed for different compressive forces.

1.3.1. Sample preparation

Each sample is immobilized by means of a graphite sheet on a metal sample plate and placed in the evaporator for 15 to 30 minutes to cover the sample of palladium vapors in order to make it conductive

1.3.2. Study method at SEM

It involves sweeping the surface of a sample using an electronic probe.

Its operation is based on the use of the thermoelectronic gun. An electron beam is produced by the vacuum heating of a Tungsten filament or Lanthanum Hexa-boride cathode. It is attracted by an anode and accelerated by an acceleration voltage to come, after having passed through electromagnetic lenses and deflection coils, to strike the surface of the sample by scanning it along two perpendicular axes. In contact with the incident beam, the sample re-emits a certain number of signals of different energies. These analyzed signals provide information on the topography, the chemical composition of the sample and atomic number contrast images (Roberts and al., 1985).

1.4. Manufacture of tablets

Series of tablets containing these different starches have been made. These tablets of variable thickness and constant weight 400mg ± 5mg were prepared by direct compression on a hydraulic press (SPEAC England) under increasing pressures: 0.5; 1; 2; 3; 4; 5 tons. Several other batches of tablets were made in this study according to the same protocol for experimental purposes.

1.5. Study of starch behavior during compression: hardness and densification

A batch of 5 tablets per type of pressure and series of starch was made. These different batches of tablets were subjected to a hardness test (pressure resistance) using ERWEKA Pharmatest PTB 301 (Hamburg-Germany). The hardness is expressed in Strong-Kobbs units. The thickness of the tablets was also determined by means of a digital caliper for each compressive force and each type of compressible starch. The starch samples were stored at room temperature in the laboratory under conditions of normal relative humidity of 25 ° C.

1.6. Method for determining the specific surface area and porosity

A tablet sample from each batch for starches studied and of known weight was subjected to the study of porosity. The measurements were carried out by adsorption of liquid gas using a COULTER SA 3100 apparatus. The tablet sample (0.04-0.07 g) was degassed under vacuum at a temperature of 50 degrees for 30 minutes. During the analysis, the pressure of the adsorbate used is gradually increased and the amount of adsorbed gas is measured as a function of the relative pressure. The adsorption is carried out at 77 degrees Kelvin (bath temperature of liquid nitrogen under atmospheric pressure). The specific surface is calculated according to the method of BRUNAUER, EMMETT and TELLER (BET) reported by SALHI (Salhi and al., 1987) from the values of the relative pressures of between 0.05 and 0.2. The volume pore size distributions are calculated according to the method of BARRETT, JOYNER and HALENDA (Salhi and al., 1987). The pore volume is obtained by converting the amount of adsorbed gas to the relative pressure 0.99 to the corresponding volume of the liquid adsorbate.

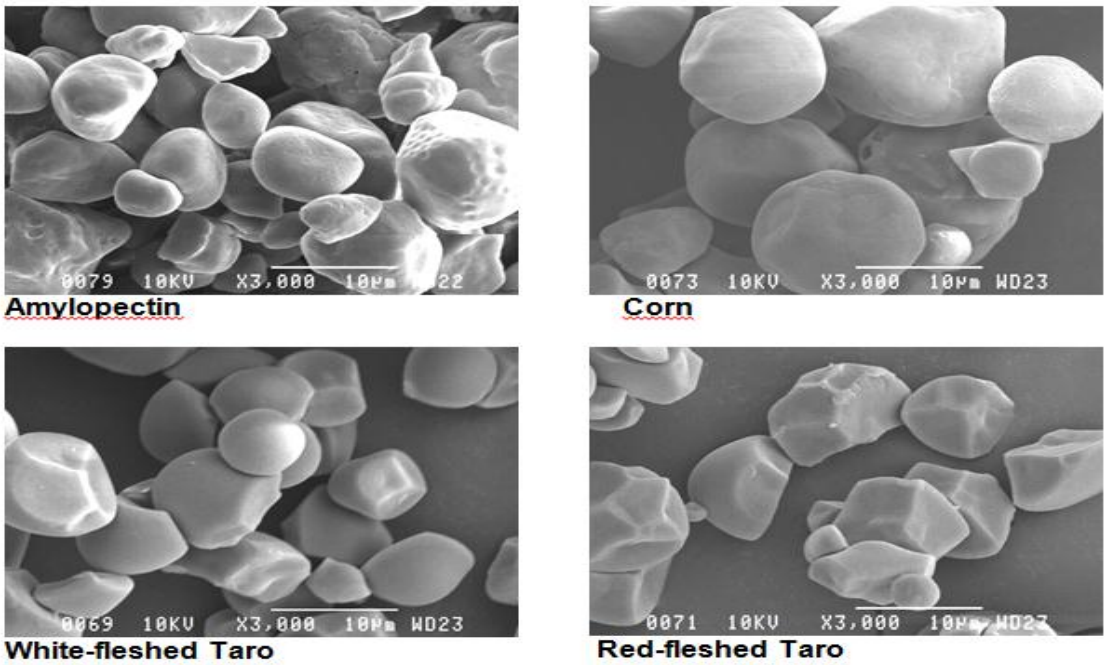
2. RESULTS AND DISCUSSIONS

2.1. At the powder level

The photographs of the grains obtained by scanning electron microscopy are presented in **Picture 1** and are characteristic of the type of material.

- Corn grains (CG) have a globally spherical shape with a polygonal tendency with well-defined edges. The size of the grains is generally between 5 and 15 microns with a predominance of small grains.
- Amylopectin (AM) grains are actually a variety of corn starch kernels with high amylopectin content. The

shape is globally polygonal but we note that the edges are rather rounded. The grain size is usually between 5 and 10 microns.
- The starch grains of the white-fleshed Taro variety (WT) have tetrahedral to polygonal shapes often rounded at the base. Grain size is usually in the range of 5-15 microns, with predominantly small grains.
- However, for the red flesh (RT) cultivar, the grains are of polygonal and elongated shape with strongly marked edges. The size is generally between 5 and 15 microns.



Picture 1: Scanning microscope photographs of different starch grains.

2.2. At the level of compression

2.2.1. Evaluation of the hardness of the tablets (Figure 1)

The starches were compressed in their natural state that is to say without undergoing gelatinization and without addition of other excipient which could have modified the natural characteristics of the materials. The goal here is to better understand the intrinsic properties of materials and not to optimize a formulation.

The other starches have shown a certain compressibility which allows their use as a diluent in unitary solid pharmaceutical forms. However, it appears that the relative humidity factor strongly conditions the results of hardness and even compressibility. Indeed, for a controlled relative humidity of 75% ± 1%, the starch gives tablets of acceptable hardness (**Callahan and al., 1982**). Subsequent work should therefore take this phenomenon into account and the starches will be stored under different conditions of relative humidity, then compressed and evaluated immediately to determine the optimum conditions for the use of our excipients.

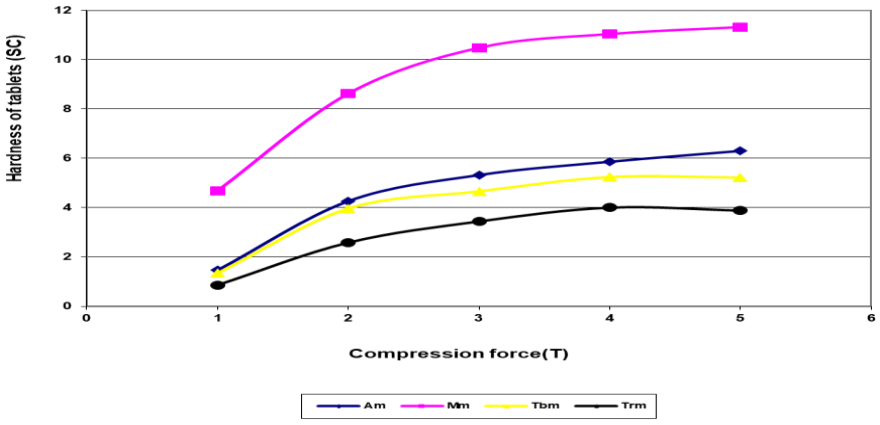


Figure 1: Relationship between tablet hardness and compressive strength for different compressible starches.

2.2.2. Study of densification and particulate rearrangement

The Heckel plots (Figure 2) show that white and red Taro starches are still rearranged at high pressures while cornstarch and amylopectin starch (Sigma Company) no longer show rearrangement beyond a compressive force greater than 2 tons. This suggests a lower elasticity of Taro starches which is a technological advantage (Guo and al., 1999).

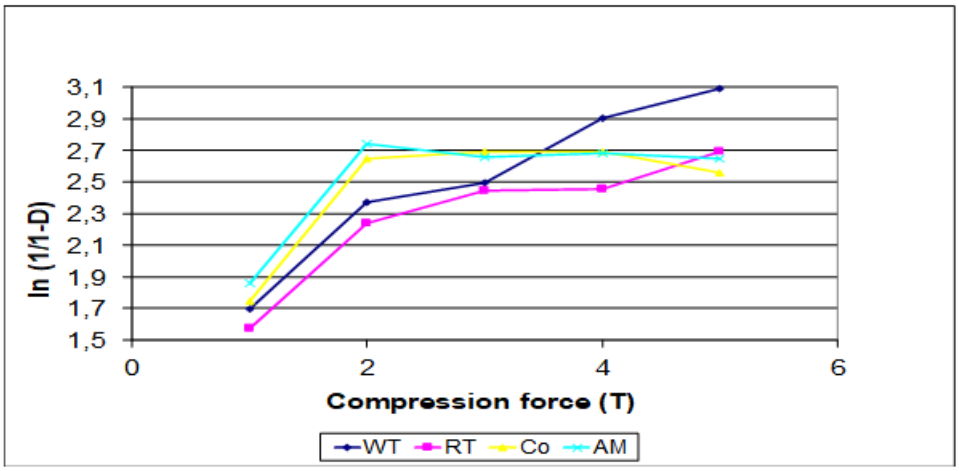
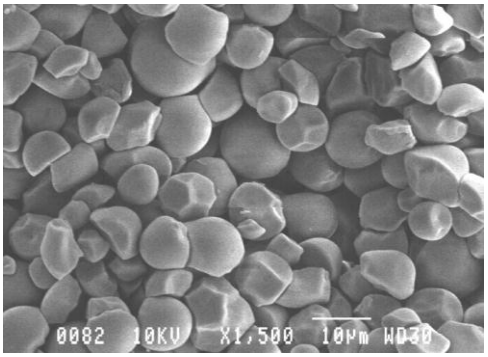
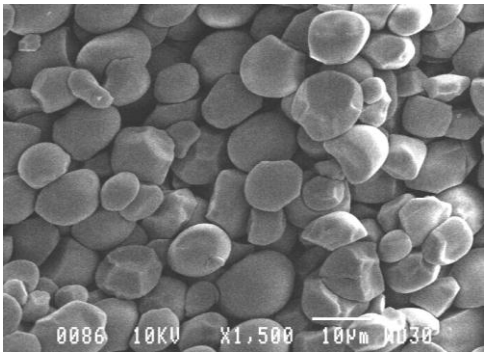


Figure 2: Heckel plot.

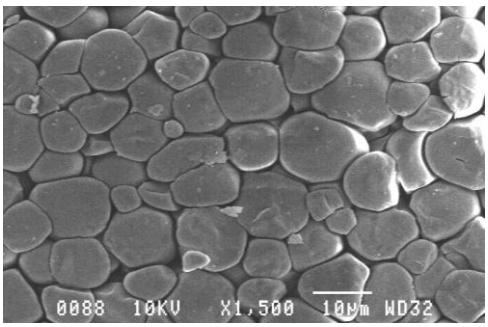
The rearrangement of grains under compression can also be observed by scanning microscopy. This analysis was performed for white-fleshed Taro (WT) and Red (TR) starches, as well as the comparison products, namely corn starch and the commercial variety amylopectin. By way of example, the photomicrographs of Taro Blanc and amylopectin, compressed at different compressive strengths, are shown in Picture 2.



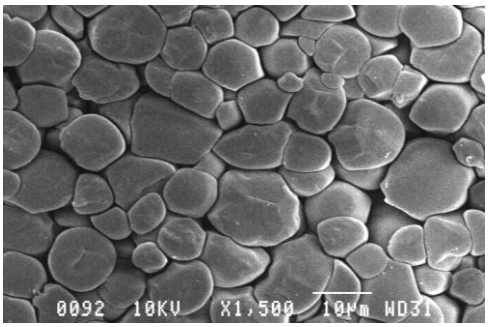
White-fleshed Taro-0,5 T



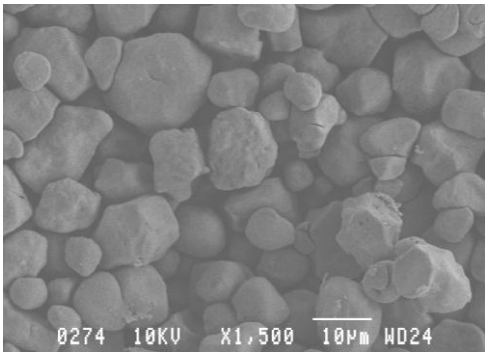
White-fleshed Taro-1,0 T



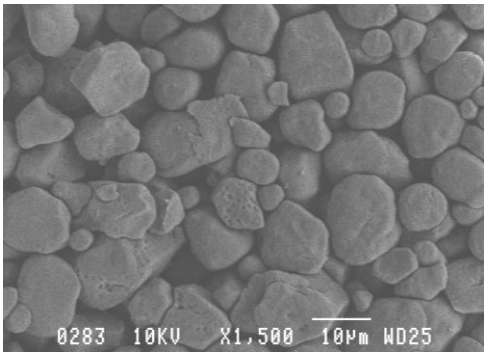
White-fleshed Taro-2,0 T



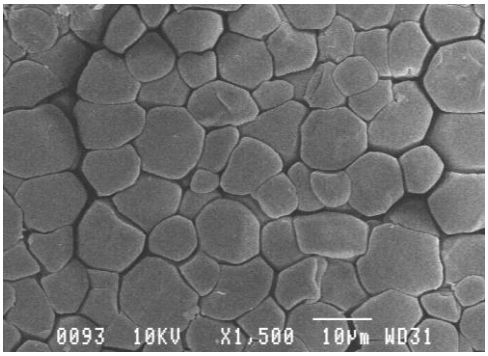
White-fleshed Taro-3,0 T



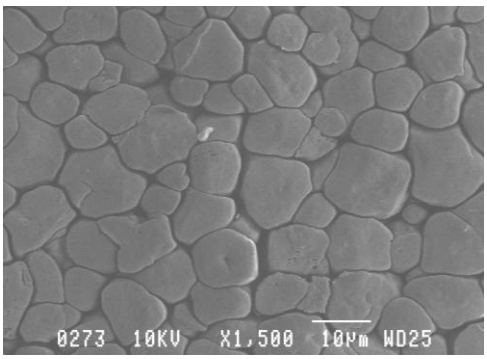
Amylopectin-0,5 T



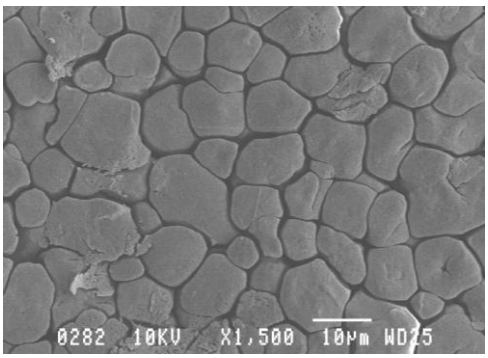
Amylopectin-1,0 T



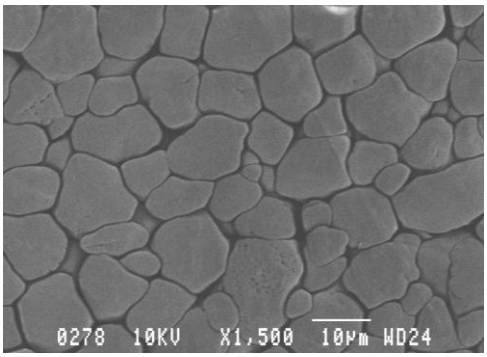
White-fleshed Taro-5,0 T



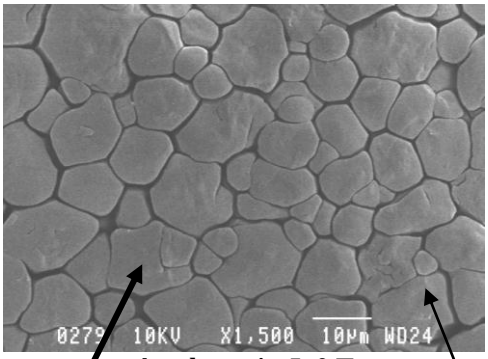
Amylopectin-2,0 T



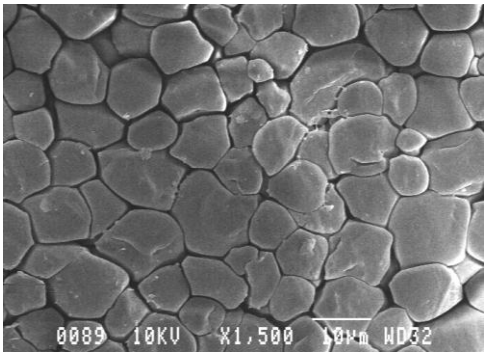
Amylopectin-4,0 T



Amylopectin-3,0 T



Amylopectin-5, 0 T



White-fleshed Taro-4,0 T

Picture 2: Scanning microscope observation of the surface of white-fleshed Taro starch tablets and corn with high amylopectin content showing particulate rearrangement under compression.

Densification and gradual granular deformation are observed as a function of the increase in compressive strength for all the starches.

It appears that forces less than or equal to one ton (1T) do not provide sufficient rearrangement, which is also reflected in the Heckel and hardness values.

Both varieties (WT and AM) show quite similar hardness curves, but different Heckel plots. It is found that amylopectin no longer shows differences in the surface structure of the tablets between 1 and 5 tons, which is in relation with the Heckel plot which shows a stabilization of the density after 2 tons. The white-fleshed Taro shows an evolution of the density, but also the deformation of the grains when increasing the

compression force of 2 to 5 tons. Indeed, the grains lose their rounded edges to adopt a more pronounced polygonal structure. This change is reflected in the Heckel plot which reveals a rearrangement process that continues beyond 2 tons.

Despite the different rearrangement, this is not marked in hardness values. However, the hardness also depends strongly and cohesion phenomena which can explain this lack of correlation. Their different water absorption capacities can also explain these results since water can play the role of plasticizer within grains (Ritala and Virtanen, 1991).

Finally, one can also realize the significant porosity that exists within these tablets especially as the starch grains retain their individuality and do not fuse for these compression forces. This explains the relatively low hardness values of starches, a phenomenon already described in the literature (**Couvreur and al., 1976**). Some fractures are however visible for high compression forces.

Porosity has been measured by gas adsorption, but the pore dimensions that can be measured by this apparatus are up to 100-200 nm. However, the microphotographs show that the pores are of much larger sizes. A mercury intrusion porosimeter will have to be used later to validate our hypotheses even if our results already show a modification of the pore size distribution during the increase of the compression.

3. CONCLUSION

The aim of the work was to characterize starches extracted from cultivars of taro species grown in Côte d'Ivoire and to compare them with varieties of corn starch. Various techniques of physical and technological characterization were successfully addressed during this study (image analysis, behavior during compression as well as determination of specific surface area and porosity).

The exploratory study of these properties allowed us to consider taro starches (white and red cultivars) as valid diluents for pharmaceutical tablet formulations.

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