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Full Length Research Paper

Stability of yam starch gels during processing

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To evaluate their aptitude to be used as functional ingredients, twenty one varieties of yam starches of Côte d'Ivoire were submitted to different technological stress such as high temperature treatment, long term freezing and refrigeration, high speed shearing and acidic treatment, in comparison with commercial modified starches. The gel of "kangba" starch (*D. Cayenensis-rotundata*) is the most stable during thermal processing. The cultivar "Daminangba" (*D. alata*) which present the clearest gel (63 % of clarity) is also the most stable during refrigeration with a low syneresis (26%) at 4°C.The "*Esculenta 7*" cultivar (*D. esculenta*) shows the weakest value of syneresis at -20°C. The gel of *D. dumetorum* species is the strongest under acidic condition with 8% of viscosity decrease from pH 7 to pH 3, whereas the "Bodo" cultivar (*D. alata*) gel shows good resistance to shearing with 31% fall of viscosity from 160 rpm to 900 rpm on the RVA. The "Sopèrè", "Lopka" and Kponan cultivars (*D. Cayenensis–rotundata*) present the strongest viscosities in all the technological treatments.

Key words: Yam starch, modified starch, functional properties, gel stability.

INTRODUCTION

Starch is one of the most important natural organic compounds, abundant in nature. It is found in the roots or fruits (Duprat et al., 1980; Buléon et al., 1990). The most common sources of food starch are corn, potato, wheat, tapioca and rice (Woolfe, 1992; Alexander, 1995; Ostertag, 1996; Wheatley et al., 1996; Henry and Westby, 1998). Developed countries (Canada, USA, Europe and Japan) have 77% of the global starch market (Sansavani and Verzoni , 1998).

The food sector consumes 55% of world production versus 45% in board industries, textile, adhesive, glue and pharmaceutical products (De Cock, 1996). In foodstuffs, starch is used to influence or control such characteristics as, aesthetics, moisture, consistency and shelf stability. It can be used to bind, expand, densify; clarify or opacify, attract or inhibit moisture. It is also used for different textures such as stringy texture, smooth texture or pulpy texture, soft or crisp coatings, and to stabilize emulsions (Luallen, 1985; Swinkels, 1985; De Cock, 1996).

Nevertheless the native starches exhibit some disadvantages certain in industrial applications. The native starch granules hydrate easily, swell rapidly, rupture, lose viscosity, and produce weak bodied, very stringy and cohesive pastes. Chemically modified starches have thus been designed to respond to industrial demand. The reticulation creates some decking among molecules, reinforcing the cohesion of the starch grain and increases its resistance to high temperature treatments (sterilization), mechanical shearing (extrusion), to acidic treatment and its stability also during freeze/thaw cycles. The stabilization by substitution of a chemical grouping (oxidation, esterification, and etherification) avoids the reorganization among the molecules after cooking. It then limit the risks of syneresis and delays or avoids the retrogradation.

Nowadays, consumers want to see more "natural" and "healthier" industrial products manufactured "without chemical processing" on the market. Some previous works show that the native starches gels of cocoyam (X. *sagittifolium*), plantain (M. *paradisiaca*), yam (*Dioscorea spp*) are resistant to sterilization (Dufour et al., 1996). Others natives starches gels are resistant to high

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shearing, like maize and wheat (Howling, 1980), quinoa and amaranth (Praznick et al., 1999). Starche gels with resistance to syneresis include tapioca (Varavinit et al., 2000), waxy maize (Yuan and Thompson, 1998), and waxy sorghum (Howling, 1980). The millet and quinoa's starches gels are described to be stable under acidic condition (Varavinit et al., 2000).

Publication on yam starches accounts is less than 1% of the total information available for food science and technology abstracts, and the food intelligence databases (Satin, 1998). The *Dioscorea* genus has a large biological diversity including more than 600 species worldwide (Hamon et al*.,* 1997). It has also a world production with more than 37 millions tonnes/year (Fao, 2000). This genus appears nowadays as a source of native starches whose functional characteristics, if sufficiently exploited, could find some applications in food ingredient industry.

The purpose of this study was to evaluate your starches for use such as in foods ingredients. This work is aimed at characterizing the functional properties of 21 varieties of yam starches from Côte d'Ivoire. Subjected to different technological stress including sterilization, refrigeration and long-term freezing, mechanical shearing, and acidic treatment.

MATERIALS AND METHODS

Starches

Twenty-one natives starches are extracted, as described in a previous work (Amani, 2002), from four species of yam tubers: five cultivars (cv) of *Dioscorea alata*, eleven cultivars of *Dioscorea cayenensis-rotundata* complex, one cultivar of *Dioscorea dumetorum*, and four clones selection of *Dioscorea esculenta***.**

Standards starches

In order to make some comparative studies, some native starches as well as one commercial starch were included in the experiments. Native starches are extracted from cassava roots (*Manihot esculenta*) and Macabo tubers (*Xanthosoma sagittifolium*) as previously described (Amani, 2002).

Normal maize starch (*Zea maïs*) was obtained from the National starch and Chemical Company (U.K), potato starch (*Solanum tuberosum*) was from Roquette & Frères (France). Colflo 67 (E1422) from National starch and chemical Company (U.K) is obtained by reticulation/stabilization. Purity HPC (E1422) from National starch and chemical Company (U.K) is obtained by reticulation/stabilization. Novation 2300 is a physically modified waxy maize starch from National starch and chemical Company (U.K). Novation 3300 is a physically modified cassava starch from National starch and chemical Company (U.K).

Amylose determination

Amylose content was measured using differential scanning calorimeter according to Mestres et al. (1996). All analyses were performed in duplicates and mean values are calculated.

Differential Scanning Calorimetry (DSC)

The DSC is performed on a Perkin Elmer DSC 7 device (Perkin Elmer, Norwalk, CT, USA) using hermetic inox pans. The duplicate sample pan (10-11 mg of starch and 50 µl of lyso-phospholipid 2% w/v in water) and the reference pan (50 ml of ultrapure water) are heated from 25°C to 160°C at a scanning rate of 10°C/min, held for 2 min at 160°C, and cooled to 60°C at 10°C/min. The enthalpy of gelatinization (ΔH) and the onset temperature (To) of each sample are then determined.

Determination of paste clarity

The procedures of Craig et al. (1989), Zheng et al. (1998) were used for determination of starch paste clarity. 1% dry basis aqueous dispersions of starch were boiled at 100°C for 30 min at constant stirring, and then transferred to plastic tubes and stored at 4°C for 4 weeks. The transmittance was measured at 620 nm using a spectrophotometer (spectronic 20 D+) every week, obtaining duplicate readings per sample.

Acid resistance

The starch sample (7%, w/v) was dispersed either in a phosphate buffer (2M pH 7) or in a citrate-phosphate buffer (2M pH 3). Using a Rapid Visco Analyzer (RVA) model 3D (Newport Scientific, Narrabeen, Australia). Viscosity was recorded using the following temperature profiles: (a) holding at 30°C for 1 min, heating from 30°C to 90°C at 6°C/min, and (b) holding at 90°C for 5 min, and then cooling to 50°C at 6°C/min with continuous stirring at 160 rpm. Final viscosity at pH 3 (VpH 3) and at pH 7 (VpH 7) were measured and the acidity effect is defined as the ratio VpH 3/VpH 7.

Shear stability

Shear stability is evaluated using the RVA on 7% aqueous starch suspensions, and final viscosity was recorded following temperature profile previously described at 160 rpm (V_{160rpm}). The experiment was repeated following the standard profile from 30°C to 90°C at 160 rpm, then held for 5 min at high shear rate (960 rpm) and cooled to 50°C at 160 rpm as the standard profile. Shear effect is calculated as the ratio of a final viscosity after shear stress (V_{960rpm}) to that of the standard condition: V_{960rpm}/V_{160rpm}. Experiments were in duplicates and mean values were calculated.

Resistance to temperature

28 g of gel sample were prepared 4%(w/w, pH_7) using RVA. The experiment is stopped after 12 min at 90°C according to the profile previously described. Viscosity is measured (V_{30}) once the gel was cooled to 30°C using Haake viscotester VT550(Germany) at $140s^{-1}$ shear rates. The experiment was repeated on the same sample after the sterilization at 121°C for 1 h, then the gel was cooled to 30°C, and the viscosity was measured as previously described (V_{121}). The temperature effect is calculated according to the formula: V_{121}/V_{30}.

Freezing and cold stability

Pastes are prepared by cooking starch slurries (4%, w/v) in water.10 g of starch paste sample were frozen in a plastic tube at –20°C, and others held at 4°C (two tubes per sample). All tubes were stored for 8 weeks. Freeze and cold stabilities were evaluated by

Table 1. Physico-chemical properties of standard starches.

Samples	Botanical name	Amylose(%)	Gelatinization Temperature (°C)	Enthalpy of Gelatinization (Joule. g^{-1})	Clarity (%)
Native starches					
Maize	*Z. mais*	27.2	65.3	11.9	27.0
Waxy maize	*Z. mais*	0.0	61.6	16.0	60.0
Potato	*S. tuberosum*	23.1	60.0	16.3	96.1
Cassava	*M. esculenta*	19.5	64.3	16.2	54.1
Macabo	*X.Sagittifolium*	26.6	76.5	15.3	26.2
Modified starches					
Colflo 67	********	0.0	62.4	15.0	21.0
Purity HPC	********	0.0	64.0	15.5	16.8
novation2300	********	0.0	60.3	15.5	19.2
novation3300	********	21.4	62.3	14.0	20.2

measuring the percentage of syneresis by centrifugation at 2660 g for 30 min. after thawing in water bath at 50°C for 90 min for frozen sample and storage at ambient temperature for 60 min for the 4°C sample respectively, according to Eliasson and Kim (1992).

Statistical analysis

One way analysis of variance (ANOVA) and principal component analysis (PCA) were performed using statistica software (version 6.0). The variables are the physico-chemical and the functional properties (viscosity at 30°C, viscosity at 131°C, RVA pH3, RVA pH7, RVA shearing shear effect, heat effect, pH effect, and syneresis after freezing and syneresis after refrigeration).

RESULTS

Characterization of standard starches

The standard starches are classified into two groups: the natural standard starches with high amylose content (about 26%) except the waxy maize starch (0%) and the tapioca starch (19%); and the modified standard starches with zero amylose content except the novation 3300 (Table 1). They were distinguished by a uniform enthalpy of gelatinization near 15 Jg^{-1} and a gelatinization temperature of 62°C except the macabo starch (76°C). The natural gels of potato, waxy maize and cassava starches are very clear (96.1; 60.0 and 54.1% of transmittance respectively), although the maize and macabo starch gels are less clear with around 26% of transmittance. The modified starches gels have low clarity according to their transmittance range from 16.8 to 20.2% (Table 1).

Resistance to syneresis

The syneresis is very high between zero and one week and becomes stabilized from the first to the 8th week hence the syneresis average was calculated between one and eight weeks (Table 2). The natural standard starches gels with low amylose content (cassava and waxy maize) appear as most resistant to syneresis after refrigeration and freezing with values varying between 0 to 4% of syneresis whereas the macabo starch gel with high amylose content has a syneresis of 38% (Table 2). The two chemically modified starches gels (Colflo 67 and Purity HPC) show less syneresis with respective average of 20% and 35%. Among commercial standards, the physically modified starches gels (Novation 2300 and Novation 3300) have both high syneresis after refrigeration and after freezing – thawing (~ 44% of syneresis) (Table 2).

The yam starches gels can behave slightly different in case of refrigeration or freezing. When they are stored at 4°C, they show a mean rate of syneresis of 41 ± 11% (Table 2). The analysis of the variance (ANOVA) does not show any significant difference between yam species. However, gaps rather quite significant are noticed between cultivars. The daminangba cultivar of *D. alata* starch gel is the most resistant to refrigeration (26% of syneresis) whereas those of kangba and frou which belong to *D. Cayenensis-rotundata* complex showed highest rates of syneresis (59%) after refrigeration (Table 2).After freezing (-21°C, 4 weeks), the yam starches gels present a higher syneresis than after 4°C refrigeration (51 ± 10%). The ANOVA gives two homogeneous groups statiscally different at p-< 0.05. The groups presenting high syneresis (~54%) include the *D. dumetorum*, *D. alata* and *D. cayenensis-rotundata* starches gels while the group which present less syneresis (~ 39%) is represented by the *D. esculenta* cultivar's starches gels. The kangba (*D. cayenensis-rotundata*) starch gel has the lowest resistant to syneresis after freezing (65% of syneresis) esculenta 7 (*D. esculenta*) starch gel distinguish itself as the most resistant of yam starches gels after freezing,with 30% of syneresis (Table 2).

Table 2. Effect of pH, shearing, stérilisation and freezing on starches gels.

pH Effect	Vf RVA Shearing (RVU)	Shear effect	Viscosity before sterilization (mPa.S)	Viscosity after sterilization (mPa. S)	Heat effect	Syneresis after freezing (%v/v)	Syneresis after refrigeration (%v/v)
0.72	144	0.69	328	265	0.81	37	35
0.64	102	0.49	219	272	1.24	57	26
0.78	100	0.62	318	273	0.86	62	28
0.62	85	0.39	234	242	1.03	58	38
0.64	75	0.38	364	263	0.72	53	36
0.68^{b}±0,07	**101^{a}±26**	**0.51^{a}±04**	**293^{a}±63**	**263^{a}±13**	**0.93^{ab}±0.21**	**53^{a}±10**	**32^{a}±5**
0.69	102	0.45	197	292	1.49	55	28
0.64	56	0.33	99	275	2.77	61	55
0.61	90	0.36	208	253	1.21	58	59
0.84	75	0.67	50	288	5.75	65	59
0.69	110	0.56	172	261	1.52	58	48
0.70	119	0.65	148	298	2.01	52	32
0.66	79	0.33	242	202	0.83	53	57
0.66	107	0.37	232	341	1.47	45	34
0.68	94	0.38	311	261	0.84	32	51
0.63	118	0.45	142	348	2.44	57	30
0.68	105	0.34	301	378	1.26	55	44
0.68^{b}±0.06	**96^{a}±19**	**0.45^{a} ± 0.13**	**191^{b}±80**	**291^{a}±50**	**1.96^{a}±1.40**	**54^{a}±9**	**45^{a}±12**
$0{,}92^{a}$	**25^{b}**	**$0{,}34^{a}$**	**61^{b}**	**115^{b}**	**$1{,}88^{a}$**	**56^{a}**	**51^{a}**
0.91	48	0.47	167	55	0.33	43	36
0.69	53	0.44	178	70	0.39	45	44
0.79	79	0.61	76	85	1.12	39	31
0.84	45	0.50	113	42	0.37	30	40
0.81^{a} ± 0.09	**56^{b} ±16**	**0.51^{a} ± 0.07**	**133^{b}± 48**	**63^{b} ± 18**	**0.55^{b} ± 0.38**	**39^{b} ±7**	**38^{a}±6**
0,71±0,09	86±29	0.47±0.2	198±92	232±99	1,44±1,18	51±10	41±11
0.81	89	0.93	88	222	2.53	36	48
0.71	42	0.50	300	60	0.20	0	4
0.67	126	0.61	851	324	0.38	65*	30*
0.77	100	0.80	363	214	0.59	4	0
0.64	67	0.52	274	116	0.42	38	38
0.88	245	0.86	219	274	1.25	20	21
0.84	245	0.95	103	305	2.98	31	39
0.74	179	0.85	36	170	4.70	42	48
1.41	49	0.96	11	100	9.11	41	45

Gel clarity behaviour during storage at 4°C

The clarity of all species of yam starches gels stored at 4°C decreases significantly during the first week then slowly beyond afterwards (Figure 1). On the other hand, the clarity of cassava starch gel which was initially high (54% of transmittance) falls slowly until the third week (50% of transmittance), and then rapidly until reaching a value lower than 40% after 4 weeks of conservation at 4°C. The natural waxy maize or chemically modified (purity HPC) starches gels present a kinetic of opacification identical to the yam starches gels at the first week, but those clarities are more stable beyond the first week.

Resistance to sterilization

In general, yam starches gels exhibit a rising viscosity during sterilization, which goes on average from 198 mPa.s before sterilization to 232 mPa.s after sterilization with a rising factor of 1.4 (Table 2). The ANOVA among yam species show no statistical difference as for heat effect. Nevertheless the most thermo stable cultivar's belong to *D. cayenensis-rotundata* and *D. dumetorum* species. Kangba from the complex *D. Cayenensis-rotundata* starch gel presents the highst viscosity after autoclaving (heat effect~6) whereas esculenta 154 belonging to *D. esculenta* starch gel resists the least with a heat effect of 0,33 (Table 2).

Among the natural standard starches gels, the one

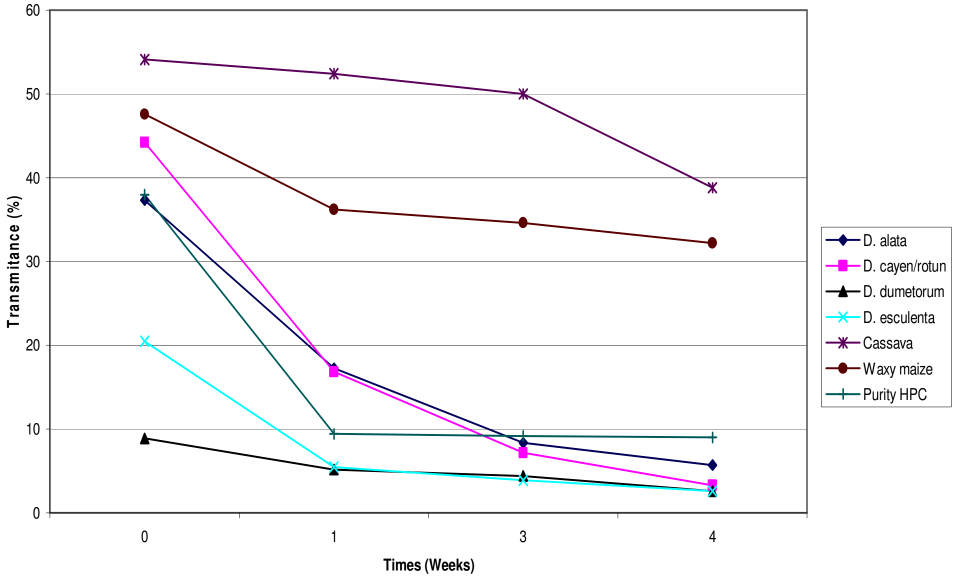

Figure 1. Evolution of clarity of starch gel during storage at 40°C.

which present the higher viscosity after sterilization is the maize starch gel (heat effect of 2.53). Whereas on the opposite side the waxy maize starch gel falls to five times of viscosity (from 300 mPa.s to 60 mPa.s) after being thermically treated (Table 2). All the modified starches gels increase their viscosity with temperature. The Novation 3300 comes in first position followed by the Novation 2300 and the purity HPC (Table 2). But the gels which present a strong viscosity after thermal treatment are first the yam starches from *D. cayenensis-rotundata* complex, notably the cultivars Sopèrè Lokpa and kponan (378, 348 et 341 mPa.s respectively). Then comes the potato starch among the natural standard starches (324 mPa.s) and purity HPC (305 mPa.s) among the modified starches.

The pH effect

The yam starches gels are sensitive to acidic condition. The fall of viscosity at pH3 runs to 31% on average. Two homogeneous groups are distinguished at $P< 0.05$. The starch gel with a strong stability in acid area (~85% of stability) summing the species of *D. dumetorum* and *D. esculenta* and the less resistant gels (68% of stability) which are the *D. alata* and the *D. cayenensis-rotundata* species together (Table 2). Among the natural standard starches gels, the maize starch is the one which resists the most to acidic condition with a stability rate of 0.81% that is to say viscosity fall of 19% in acid area. On the other hand among the modified starches all resistant to acidic condition. The increase of viscosity in acidic condition which goes up to 41% for the Novation 3300 can be noticed specially (Table 2).

Effect of shear stress

The chemically modified (Purity HPC and Colflo 67) and physically modified (Novation 3300 and 2300) starches gels are those which are the most resistant to high shear treatment (Table 2). Among the natural standard starches gels, only the maize starch resists to shearing with only 7% of viscosity fall while the waxy maize is the less stable to shearing (50% of viscosity fall). The yam starches gels are also sensitive to shearing (Table2). The shear effect (47±12% of stability) shows a very low significant variation among the different yam species. The *D. dumertorum* starch gel is the most sensitive (66% of viscosity fall after shearing at 960 tr/min), whereas the bodo cultivar starch gel is the yam which resist the most to shear stress with 31% of viscosity fall. These values are higher than those of the modified standards, which are more stable to the shear stress (Table 2).

DISCUSSION

Principal component analysis

The principal component analysis evaluated on the 21 yam cultivars from 10 variables (physico-chemical and functional properties) gives 3 principal components. The principal component 1 (PC1) (showing 44% of the variation) emphasizes essentially the variation due to viscosity and pH effect. Viscosity and pH effect are correlated negatively. The principal component 2 (PC2) (showing 21% of the variation) described essentially syneresis after freezing and autoclaving effect. The last

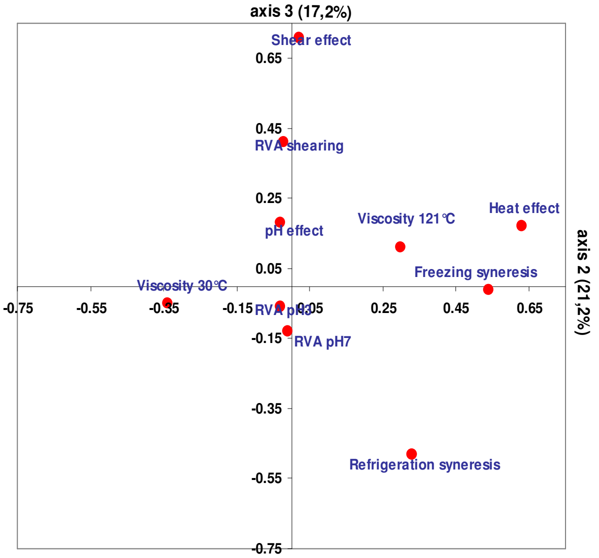

Figure 2. Loading plot of PC2 and PC3.

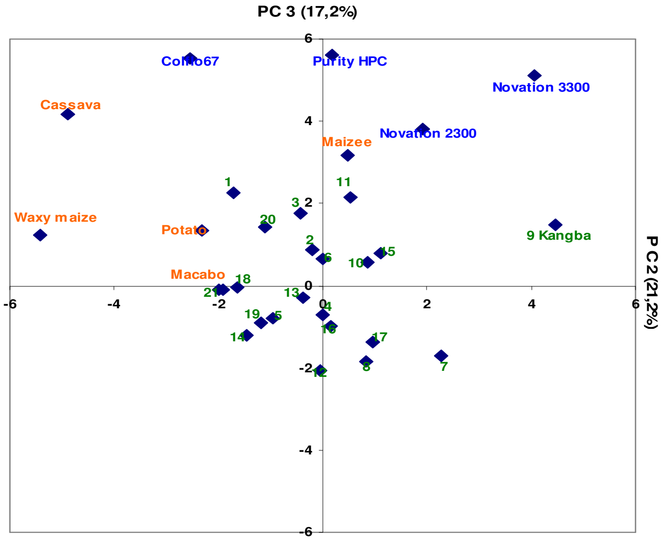

Figure 3. Score plot of principal components 2 and 3. From 1 to 5: *D. alata*, 6 to 16: *D. cayenensis- rotundata,* 17: *D. dumetorum*, 18 to 21: *D. esculenta.*

one, principal component 3 (PC3) (showing 17% of the variation) emphasizes essentially the shear effect and the syneresis after cooling. A negative correlation is found for these two variables. The last two principal components (PC2 and PC3) reveal the unity of starch gels functional characteristics and their stability during technological treatments except for acidic treatment (Figure 2).

The projection of samples on the axis 2 and 3 previously defined, lead to 4 homogeneous clusters with identical properties (Figure 3). The first group contains the chemically modified starches (colflo 67 and purity HPC), situated in the forefront of axis 3. These starches gels are stable to shear stress and to syneresis. The second group is represented by the physically modified starches gels (novation 2300 and novation 3300). These are in the middle of axis 2 and 3, they are resistant to heat treatment, to acidic treatment and to shear stress, and they also have high syneresis. The third group (cassava, waxy maize and potato) are natural standard starches gels presenting a good clarity, stable to syneresis and unstable to heat treatment. These results are in harmony with a previous work of Hoover and Manuel (1996) on the waxy maize starch syneresis and Vanavinit et al. (2000), on the cassava starches. The yam starches are in the center of axis 2 and 3. Yam starches gels are characterized by their weak stability to the technological stress (Figure 3).The maize starch is the only natural standard close to the modified starches because of its stability to shearing, acidic condition and sterilization. These results confirm those of Howling (1980) and Khun and Schlauch (1994), which previously had revealed the stability of maize to mechanical shearing. Among yam starches, only the kangba starch gel is close to the physically modified starches (Novation 2300 and 3300) gels in terms of stability to sterilization, shearing, acidic treatment and syneresis after refrigeration.

Kangba , assobayérè and lokpa starches gels are also resistant to temperature whereas *D. dumetorum* and *D. esculenta* gave gels with a strong stability in acidic condition.

The yam starches are less resistant to syneresis after long-term freezing or refrigeration. The mean value of syneresis at 4°C and -21°C and gelatinization enthalpy are correlated negatively (r = -0.47). The mean value of syneresis at 4°C and -21°C and temperature effect are correlated positively (r = +0.59). This indicates that the yam starches gels which are the less stable to syneresis are the most stable to thermal treatment. On the other hand, syneresis and low amylose content are not correlated. But up to 21%, the syneresis increases according to amylose content confirmingt the previous studies (Zheng and Sosulski, 1998; Dufour et al. 2000 and Varavinit et al. 2002). Beyond 20%, the amylose has no significant effect on syneresis. This would probably show that other phenomena occur in the syneresis. High amylose content is also linked to starch retrogradation phenomenon (Ortega-Ojeda and Eliasson 2001). So the high amylose content of yam starches would then be at the origin of their instability to the long-term freezing or refrigeration. This result is confirmed by the opacification of the yam starches gels during storage at 4°C. In comparison, gels obtained with starches having low or no amylose content such as cassava, waxy maize or purity HPC have a higher clarity. In fact Jacobson et al. (1997) showed that the storage of gels at cold temperature would accelerate the retrogradation of amylose and

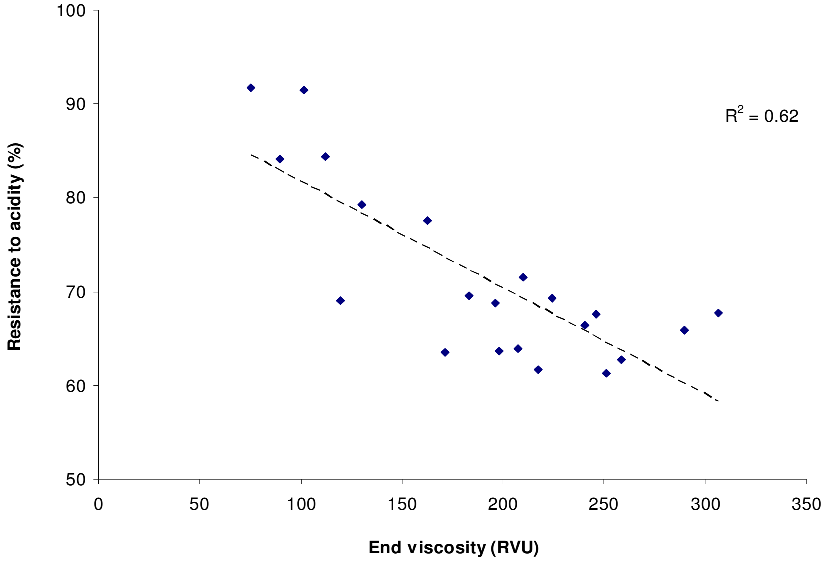

Figure 4. Relation between RVA viscosity and acid resistance of starch gels.

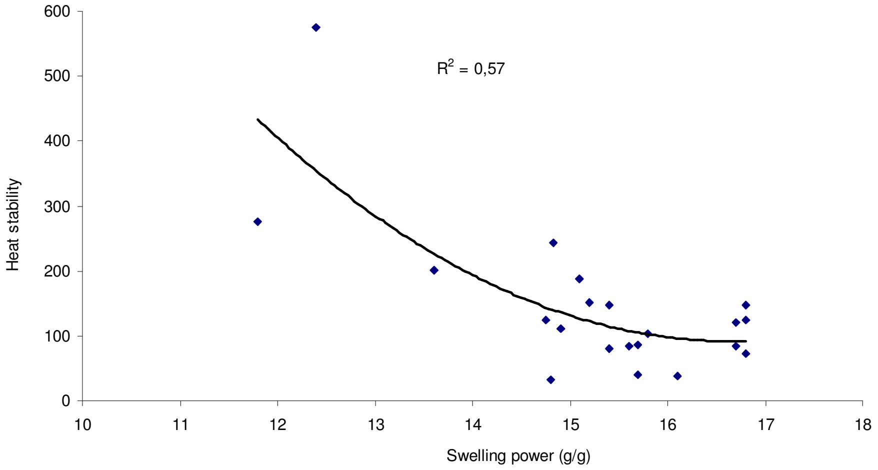

Figure 5. Relation between swelling power and heat stability of starch gels.

reduce the gel clarity.

The starch gels with a low amylose content are predisposed to acidic resistance. In fact, the stability to acidic treatment and amylose content are correlated negatively (r = -0.70). The negative correlation between acidic resistance and RVA viscosity shows that the starches gels with a high viscosity are the most sensitive to acidity (Figure 4). The case of waxy maize, which goes against the rule, could be explained by the fact that the pH effect has been measured under the shearing constraint. Furthermore, yam starches gels are also unstable to high shearing, compared to the chemically modified starches. That is probably due to a weak rate of macromolecular reticulation of yam starches. In fact Purity HPC and colflo 67have been obtained by the combination of reticulation by adipate and stabilization by acetate, with high degree of cross-bonding. This reticulation creates some decking between the macromolecules leading to an amylose network resistance to shearing and acidic treatment (Langley, 1995; De-Cock, 1996; Rôper, 1997).

The heat stability of starches gels and swelling are correlated negatively (r = -0.70) (Figure 5). The swelling power previously characterized by Amani et al (2002) indicates a stage of complete solubilization and gelation. On the other hand the resistance to sterilization and amylose content are independent (r = 0.23). Nevertheless amylose content and viscosity after sterilization are strongly correlated (r = 0.88).

This study has revealed yam starches (regardless of their specie) have similar reactions to technological stress. Only the kangba yam starch (*D. cayenensis-rotundata*) shows properties close to the modified starches. Yam starches can be used as natural component in emulsified sauces, because of their tolerance to shearing and acidity. Moreover, their stability to high temperature treatment makes them a good texture agent for manufactured products at high temperature or as a highly sterilizable product like baby foods. The kangba starch gel with those of *D. dumetorum* and of *D. esculenta* species, which are stable in acidic condition and which develop low viscosity, can be substitute for modified starches as a functional ingredient for low pH processed foods like sauces and salad dressings, and are also ideal for retorted food.

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