

Pulping of fibre sorghum in a twin screw extruder (extrudeur bi-vis).

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Summary: The Thermo-Mechanico-Chemical fractionation in twin screw extruder of pith of sorghum fibre gives a pulp with a 50% yield. Cardboard obtained from this pulp shows characteristics which can be compared to those of recycled paper.

The result leads at the fractionation of the stalk of sorghum fibre in a twin screw reactor. The pulp achieved with a 80% yield is appropriate for an application in cardboard packaging.

The screw profile and the configuration of the twin screw reactor are adapted with each material (pith and stalk of sorghum fibre).

Experimental designs were employed to study the influence of the sodium hydroxide concentration and temperature.

Key words: Fibre Sorghum, fractionation, paper, pulp, twin-screw extruder

Fabrication de pâtes à papier de Sorgho à Fibre par le procédé extrudeur bi-vis

Résumé: Le fractionnement Thermo-Mecano-Chimique en réacteur bi-vis de la moelle de sorgho donne un rendement en pulpe (raffinat) de 50 %. Le papier carton fabriqué à partir de cette pulpe présente des caractéristiques comparables à celles du papier recyclé.

Ce résultat a conduit au fractionnement de la tige entière de sorgho à fibre dans un réacteur bi-vis. Les pâtes obtenues avec un rendement de 80 % conviennent parfaitement pour les applications en emballage carton.

Le profil des vis et la configuration du réacteur sont adaptés à chaque matériau (moelle ou tige entière de sorgho).

Les effets de la température et de la concentration en soude sont étudiés grâce à des modèles expérimentaux.

Mots clés : Sorgho à fibre, fractionnement, pulpe, papier, extrudeur bi-vis

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1. Introduction

The increase, in the last twenty years, of the world paper and cardboard consumption ^[1,2] together with global environmental restrictions due to timber overexploitation, have led to research on the diversification of sources of raw materials. Many investigations have been carried out on the use of annuals or of cultivated lignocellulose residues for the manufacture of paper pulp ^[3, 4, 5].

Among fibre annuals, fibre sorghum has been the centre of many studies ^[6, 7], for purposes including varietal selection and cultivation methods. ^[8, 9] The stem of the plant, which accounts for 75 % of the total weight of its aerial system, contains however an important proportion of pith (15 to 20 %). The presence of this pith has penalized its use in traditional paper industry processes ^[10] and lead to the implementation of mechanical depithing processes ^[11] as for bagasse, the fibrous residue of sugar cane. ^[12]

Concurrently, new paper pulping processes were developed and studied for lignocellulose fibre treatment of annuals: thermo-mechanical fractionation by steam explosion ^[13, 14], thermo-chemical fractionation in hydro-organic environment ^[15, 16] and thermo-mechanico-chemical fractionation in twin screw extruder ^[17, 18, 19].

Our aim here is:

- i) to study the influence of the main operating variables on the paper qualities of the pulps achieved by twin-screw fractionation of fibre sorghum ;
- ii) to suggest a process for the obtainment of paper pulps without fibre sorghum depithing.

2. Methods

2.1. Raw material

The sorghum is provided by the Company Protosemences (France). It is

crushed in a VSI electra type hammer mill (Poudenas, 47170 France). Its average composition is the following: cellulose 38 %, hemicelluloses 28 % and lignin 9.6 %. The pith fraction achieved by mechanical depithing in a Pallman grinder (PMS type Pallman Centurion Depither) is provided by the Paper Technical Centre (Grenoble, France). Its average composition is : cellulose (42.5 %), hemicelluloses (22.5 %) and lignin (17.5 %).

2.2. Twin screw extruder

Sorghum fractionation was achieved in a modified BC 45 twin screw CLEXTRAL extruder (Firminy 42702 France) with co-penetrating and co-rotating screws ^[20]. The barrel is 1.4 metre long and divided into seven 20 cm sections. Each section holds a circulating water cooling system and two openings which may be used for the injection of liquids. Four of the seven sections can be heated by induthermes. The first element of the barrel is an introduction section. It has an opening over which is placed a measuring hopper. The solid can therefore be introduced freely. The filtering section is fitted with two semi-cylindrical filters consisting of easy to dismantle perforated belts with conical holes (1 mm inlet and 2 mm outlet). The barrel is closed by a front die consisting of two 40 mm long cylindrical holes, 10 mm in diameter at its entrance and a 25 mm long cylindrical hole, 12 mm in diameter at its exit. Five kinds of elements form the profile of the screws used: T2F (Twin flight trapezoidal), C2F (Twin flight self wiping), Mal0 (Eccentric paddles), Mal2 (Bilobal paddles) assembled at 90° and C2FC (Twin flight reverse pitch self wiping), this last device situated after the filtering section enables the separation of the liquid and solid phases. The

sodium hydroxide solution is injected by means of a volumetric pump.

2.3 The examined responses

The responses which were examined are:

- the electrical intensity used by the engine (I) expressed in A.
- the dryness of the solid coming out from the reactor (MS) given in % related to the out-coming solid mass (raffinate).
- the yield of raffinate (Rd), expressed by the raffinate dry matter flow-rate/input dry matter flow-rate ratio.
- the paper qualities of the transformed fibre, measured through the mechanical characteristics of the sheets obtained through refining in a Sprout-Waldron device (Paper Technical Centre at Grenoble, France) :
 - The Breaking length (LR) expressed in km (NF Q03-002).
 - The burst index (IE) expressed in kPam²/g (NF Q03-053).
 - The tear index (ID) expressed in mNm²/g (NF Q03-001).
 - The Concora Medium Test (CMT) expressed in N (NF Q03-044) and the Ring Crush Test (RCT) expressed in N (TAPPI T818).

2.4. Doehlert's experimental designs

The effects of temperature (T) and sodium hydroxide concentration (NaOH) are studied through experimental design Doehlert's type ^[21]. The second order polynomial model linking each response to the factors is such as:

$$\eta = \beta_0 + \sum \beta_i X_i + \sum \beta_{ij} X_i X_j$$

with : η = response ; β_0 = constant term ; β_i first order coefficient ; β_{ij} corresponding to first order interaction

terms if $i \neq j$ and to the squared terms if $i = j$.

X_i is a reduced variable: $X_i = (U_i - U_i^0)/\Delta U_i$

U_i^0 is the interest point, ΔU_i the variation step. The natural variable T is noted U_1 and the NaOH variable U_2 .

The NOMROD software ^[21] was used for the determination of the coefficients of the second order polynomial equation of the model as well as for the printing of the isoresponse curves.

3. Results and discussion

Mechanical depithing of fibre sorghum through crushing and densitometric separation (Pallman depither) makes it possible to separate a fibrous fraction, the quality of which is well-known for the manufacturing of paper pulps ^[22]. The residual fraction consists of fine particles (dust, ground leaves), of transported pith and fibrous matter. It accounts for 50 % of the introduced fibre sorghum dry matter. The enhancement of its value is therefore crucial for the economic feasibility of the manufacturing process of paper pulps of sorghum origin.

3.1. Enhancement of fibre sorghum pith value

The aptitude of the pith obtained by mechanical fraction of fibre sorghum and intended for transformation into paper is examined in a twin-screw machine.

The screw profile and configuration implemented include an Eccentric paddles zone (mal0), a reverse screw after the filtering section and a front die (table 1). The roles of different screw elements have been defined in previous work ^[20,23].

The influence of temperature and sodium hydroxide concentration in the water solution injected is examined after the carrying out of experimental

design Doehlert's type (table 2). In all operating conditions, the fibrous pulps obtained led to the production of paper sheets (table 3).

The second order polynomial model equations linking each of the examined responses to the coded variables associated to temperature and sodium hydroxide concentration factors, are the following:

$$I = 12.49 - 1.06X_1 + 0.467X_2 - 0.238X_1^2 - 5.297X_2^2 + 0.268X_1X_2$$

$$MS = 35.50 - 1.869X_1 + 0.653X_2 + 2.345X_1^2 - 4.337X_2^2 + 1.362X_1X_2$$

$$Rd = 75.78 + 5.54X_1 + 1.809X_2 - 20.88X_1^2 - 13.93X_2^2 + 11.35X_1X_2$$

$$LR = 3.22 - 0.416X_1 + 0.373X_2 - 1.624X_1^2 - 0.245X_2^2 + 0.325X_1X_2$$

$$IE = 1.108 - 0.188X_1 + 0.097X_2 + 0.013X_1^2 - 0.052X_2^2 - 0.072X_1X_2$$

$$ID = 351.2 + 44.5X_1 - 16.7X_2 - 66.4X_1^2 + 11.8X_2^2 + 38.4X_1X_2$$

$$CMT = 148.2 - 21.8X_1 + 3.17X_2 + 14.6X_1^2 - 6.07X_2^2 + 12.3X_1X_2$$

$$RCT = 138.4 - 28.3X_1 - 3.11X_2 + 17.9X_1^2 - 10.3X_2^2 + 21.5X_1X_2$$

The analysis of the isoresponse curves plotted according to these models bring to the fore the following points:

- raffinate yield reaches its maximum near the centre of the experimental field (figure 1). A decrease in sodium hydroxide content, and in a smaller proportion an increase in temperature, favour the extraction of a greater proportion of pith dry matter. It should be noted that with sodium hydroxide content down to a minimum, and for an average temperature, the water content of the raffinate is minimal (figure 2), and the intensity of the power used is maximal (figure 2). The latter is directly correlated to the specific mechanical

power as long as flow rates vary little. The intensification of the mechanical processing of the fibre with little sodium hydroxide induces a better centrifugation of the raffinate and the transport of a higher proportion of thin particles (< 0.5 mm) through the filters. Extracts are indeed carrying suspended particles. However, an increase in the sodium hydroxide content and a decrease in temperature induce a greater solubilisation of the hemicelluloses. On the whole, a higher proportion of dry matter is also eliminated.

- the paper sheets' mechanical resistance to stretching, bursting and crushing after refinement in a Waldron sprout is improved by a decrease in the sodium hydroxide content when associated to :

- an increase in temperature for the breaking length (figure 3) and burst index (figure 3) ;
- or an average temperature (60°C) for the Concora medium test (figure 4) and the Ring Crush Test (figure 4) ;

- The tear index only, deviates from this orientation. The isoresponse curves reach their maximum near the centre of the experimental field (figure 5).

These results show that as far as the pith fractionation of fibre sorghum obtained by mechanical depithing in a twin screw extruder is concerned, it is the mechanical process eliminating a high proportion of fine particles which is the most advantageous regarding the mechanical qualities of the paper sheets, tear index excepted. Minimal sodium hydroxide content is then sufficient to enable, through the thermo-mechanical solubilisation of the "harmful" constituents, and the mechanical elimination of fine particles, a more efficient reorganisation of the fibrous

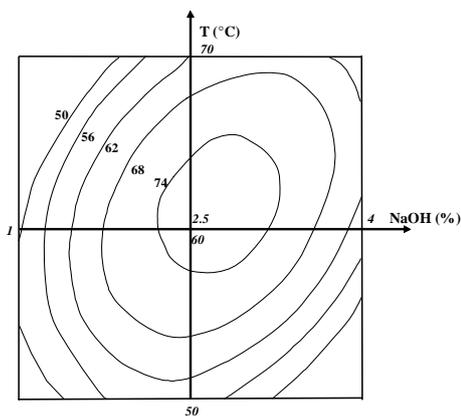


Figure 1: Treatment of pith of fibre sorghum: Isoreponse curves for the yield of raffinate Rd (%), as a function of temperature, T, and extracting solution concentration, NaOH. Operating conditions: input solid flow rate, 5.3 kg/h, screw speed rotation, 150 rpm, and input liquid flow rate, 36.2 kg/h.

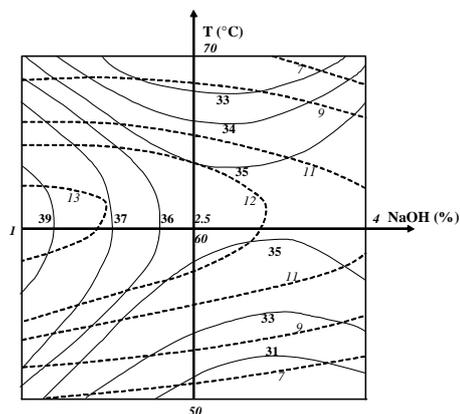


Figure 2: Treatment of pith of fibre sorghum: Isoreponse curves for the dryness of the raffinate MS (%) (line —), and Isoreponse curves for the electrical intensity I (A) (line ----) as a function of temperature, T, and extracting solution concentration, NaOH. Operating conditions: input solid flow rate, 5.3 kg/h, screw speed rotation, 150 rpm, and input liquid flow rate, 36.2 kg/h.

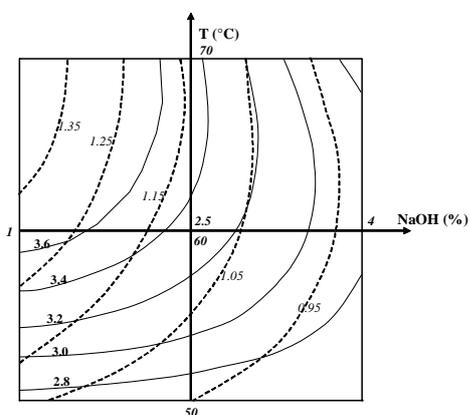


Figure 3: Treatment of pith of fibre sorghum: Isoreponse curves for the Breaking length LR (km) (line —), and Isoreponse curves for Burst Index IE (kPam²/g) (line ----) as a function of temperature, T, and extracting solution concentration, NaOH. Operating conditions: input solid flow rate, 5.3 kg/h, screw speed rotation, 150 rpm, and input liquid flow rate, 36.2 kg/h.

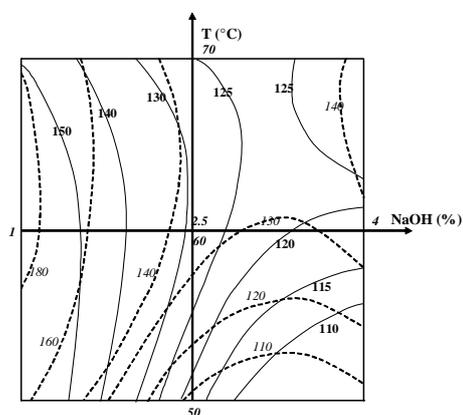


Figure 4: Treatment of pith of fibre sorghum: Isoreponse curves for the Concora Medium Test CMT (N) (line —), and Isoreponse curves for Ring Crush Test RCT (N) (line ----) are a function of the temperature, T, and the extracting solution concentration, NaOH. Operating conditions: input solid flow rate, 5.3 kg/h, screw speed rotation, 150 rpm, and input liquid flow rate, 36.2 kg/h.

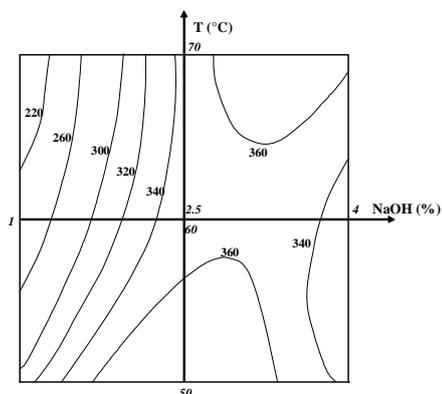


Fig. 5. Treatment of pith of fibre sorghum: Isoreponse curves for the tear index ID (mNm²/g) as a function of temperature, T, and extracting solution concentration, NaOH. Operating conditions: input solid flow rate, 5.3 kg/h, screw speed rotation, 150 rpm, and input liquid flow rate, 36.2 kg/h.

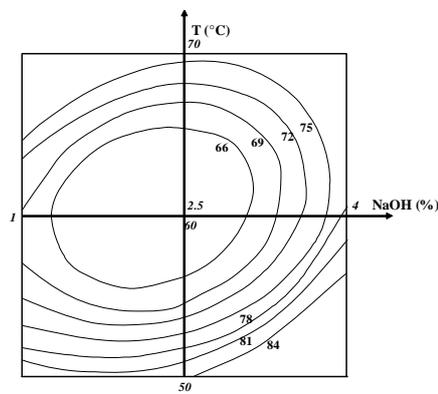


Fig. 6. Treatment of fibre sorghum: Isoreponse curves for the yield of raffinate Rd (%), as a function of temperature, T, and extracting solution concentration, NaOH. Operating conditions: input solid flow rate, 7.2 kg/h, screw speed rotation, 150 rpm, and input liquid flow rate, 31.2 kg/h.

Table IV: comparison of the mechanical characteristics of pith of fibre sorghum with waste papers pulp.

	LR (km)	IE (kPam ² /g)	ID (mNm ² /g)	CMT (N)	RCT (N)
Pith of fibre sorghum	3.66	1.34	221	187	190
Waste papers	3.68	2.62	790	169	182

Table V: Configuration and screw profile in the twin screw extruder for treatment of stalk of fibre sorghum

Zone	1		2		3						4		5		
configuration	Solid Feed				Induction Belts						filtration		Induction Belts		
Screw profile Type	T ₂ F	C ₂ F	C ₂ F	C ₂ F	Mal2	C ₂ F	C ₂ FC	C ₂ F							
Pitch (mm)	66	50	33	33	10x10	33	33	25	33	33	25	25	25	-25	33
Length (mm)	100	100	100	100	10x10	100	100	100	100	2x50	2x50	2x50	2x50	2x50	100

Table VI: Experiment matrix for the study of influence of sodium hydroxide concentration and the temperature on treatment of stalk of fibre sorghum in the twin screw extruder.

Experiment	NaOH		T	
	X ₁	U ₁ (%)	X ₂	U ₂ (°C)
S ₁	0	2.5	0	60
S ₂	1	4	0	60
S ₃	-1	1	0	60
S ₄	0.6	3.4	1	70
S ₅	-0.3	2	-0.8	52
S ₆	0.6	3.4	-0.8	52
S ₇	-0.3	2	1	70

U₁[°] = 2.5%; ΔU₁ = 1.5% U₂[°] = 60°C ; ΔU₂ = 10 °C

Operating conditions: Average solid flow rate : 7.5kg/h ; average liquid flow rate : 31.2 kg/h ; screw speed rotation :150 tr/mn

Table VII: Experiment result for the treatment of the stalk of fibre sorghum in twin screw extruder. Refiner Sprout Waldron: °SR 50.

Experi ment	I (A)	MS (%)	Rd (%)	LR (km)	IE (kPam ² /g)	ID (mNm ² /g)	CMT (N)	RCT (N)
S ₁	17	37.8	61.4	2.95	1.03	347	148	143
S ₂	13	33.7	78.9	2.75	1	392	150	118
S ₃	34	48.2	67.5	3.66	1.34	221	187	190
S ₄	12	35.9	78.4	3.54	1.19	337	147	135
S ₅	19	39.9	74.2	3.25	1.13	337	150	140
S ₆	15	35.8	82.2	2.72	0.88	363	137	123
S ₇	18	39.8	78.9	2.73	0.98	325	132	121

matrix macromolecules. Cardboard obtained from pith raffinate achieved with a 50 % yield, shows characteristics which can be compared to those of recycled paper, except for the tear index (table 4). More particularly, the pith raffinate could be mixed to other fibres to take advantage of its crush resistance properties (Ring Crush Test and Concora Medium Test). This fibre supply could as a matter of fact be provided by the fibrous fraction obtained from sorghum mechanical depithing. It should not be forgotten that a great proportion of the fine particles are the result of this crushing treatment. Direct transformation of whole sorghum (stalk) could therefore be considered highly interesting.

3.2 Transformation of whole sorghum (stalk) in a twin screw extruder

The screw profile and implemented configuration consist in a Bilobal paddles zone (Mal2), a reverse screws situated after the filtering module and a front die (table 5) The roles of different screw elements have been defined in previous work^[20,23] ..

The experiment design achieved in order to examine the influence such factors as temperature and sodium hydroxide content in the injected solution, or the experimental field, are shown in table 6. The mechanical characteristics of the pulps obtained after refining are shown in table 7.

The second degree polynomial model equations linking each of the examined responses to the coded variables, associated to the temperature and sodium hydroxide content factors are the following:

$$I = 16.52 - 0.83X_1 - 9.70 X_2 - 0.69X_1^2 + 7.22X_2^2 - 1.11X_1X_2$$

$$MS = 61.72 + 4.41X_1 + 0.653X_2 + 2.345X_1^2 - 4.337X_2^2 + 1.362X_1X_2$$

$$Rd = 75.78 + 5.54X_1 - 2.27X_2 + 11.17X_1^2 + 16.61X_2^2 - 5.76X_1X_2$$

$$LR = 2.98 + 1.60X_1 - 0.247X_2 + 0.415X_1^2 - 0.172X_2^2 - 1.26X_1X_2$$

$$IE = 1.63 + 0.68 X_1 - 6.54 X_2 - 0.105X_1^2 - 0.351 X_2^2 - 0.312X_1X_2$$

$$ID = 483.9 + 37.8X_1 + 35.8X_2 - 123.4X_1^2 - 57.9X_2^2 - 18.8X_1X_2$$

$$CMT = 151.3 + 31.3X_1 - 6.72X_2 - 9.97X_1^2 - 16.9X_2^2 - 30.1X_1X_2$$

$$RCT = 151.6 + 36.1X_1 - 6.51X_2 - 17.3X_1^2 - 10.3X_2^2 - 37.3X_1X_2$$

The analysis of isoresponse curves plotted according to these models bring to the fore the following points:

Unlike sorghum pith, the raffinate yield shows a minimum near the centre of the experimental field (figure 6). But, as in the case of pith, a low sodium hydroxide content in the injected solution induces a higher specific mechanical energy transferred to the matter and a better centrifugation of the fibre (figure 7). However, even though whole sorghum is crushed into 2 cm long strands in a hammer mill before being introduced in the twin-screw reactor, this operation does not generate as high a quantity of fine particles as in depithing.

The important effect observed in the case of pith subject to mechanical treatment conditions, when the fine particles are driven through the filter, is not so perceptible in the case of whole sorghum.

Unlike sorghum pith, it is an increase in sodium hydroxide content associated to a decrease in temperature which improve the mechanical qualities of the paper sheets (breaking length: figure 8; Concora Medium Test: figure 9; Ring Crush Test: figure 9) except for the tear index (figure 10).

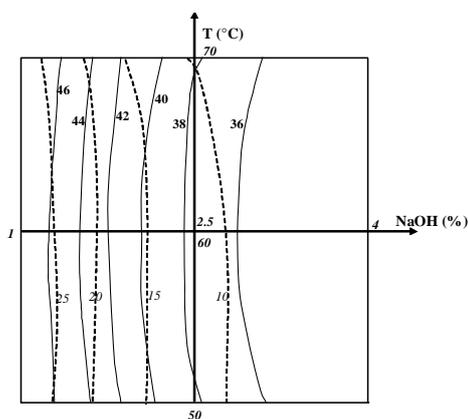


Figure 7: Treatment of fibre sorghum: Isoreponse curves for the dryness of the raffinate MS (%) (line ___), and Isoreponse curves for the electrical intensity I (A) (line ----) as a function of temperature, T, and extracting solution concentration, NaOH. Operating conditions: input solid flow rate, 7.2 kg/h, screw speed rotation, 150 rpm, and input liquid flow rate, 31.2 kg/h.

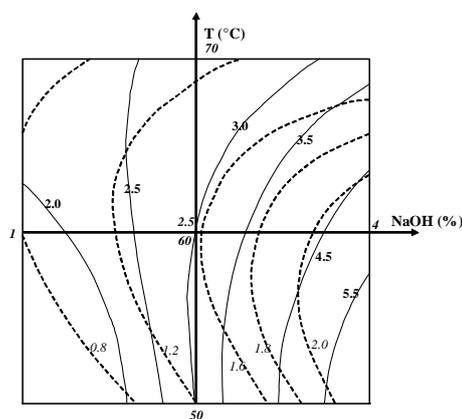


Figure 8: Treatment of fibre sorghum: Isoreponse curves for the Breaking length LR (km) (line ___), and Isoreponse curves for Burst Index IE (kPam²/g) (line ----) as a function of temperature, T, and extracting solution concentration, NaOH. Operating conditions: input solid flow rate, 7.2 kg/h, screw speed rotation, 150 rpm, and input liquid flow rate, 31.2 kg/h.

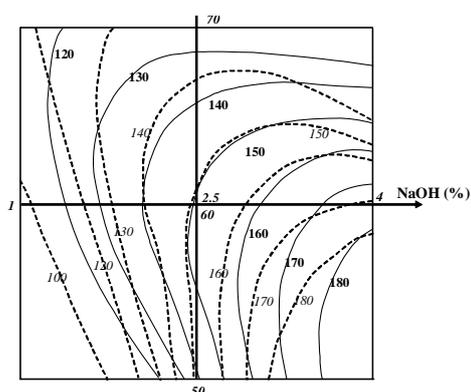


Figure 9: Treatment of fibre sorghum: Isoreponse curves for the Concora Medium Test CMT (N) (line___), and Isoreponse curves for Ring Crush Test RCT (N) (line ----) as a function of temperature, T, and extracting solution concentration, NaOH. Operating conditions: input solid flow rate, 7.2 kg/h, screw speed rotation, 150 rpm, and input liquid flow rate, 31.2 kg/h.

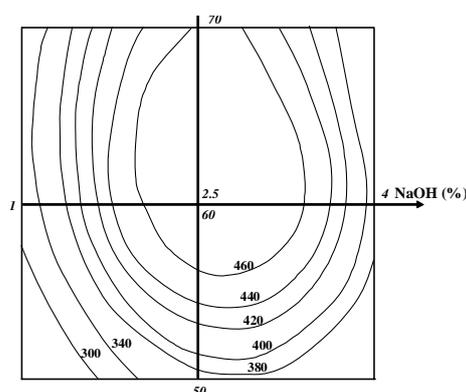


Figure 10; Treatment of fibre sorghum: Isoreponse curves for the tear index ID (mNm²/g) as a function of temperature, T, and extracting solution concentration, NaOH. Operating conditions: input solid flow rate, 7.2 kg/h, screw speed rotation, 150 rpm, and input liquid flow rate, 31.2 kg/h.

Table VIII : comparison of the mechanical characteristics of stalk of fibre sorghum pulp with or without water washing.

	LR (km)	IE (kPam ² /g)	ID (mNm ² /g)	CMT (N)	RCT (N)
without water washing	4.25	1.83	553	197	144
With water washing.	3.19	1.22	611	175	129

Operating conditions: T: 52°C ; NaOH : 3.5%

In that case, with low sodium hydroxide content, even at a high temperature, but unlike sorghum pith, thermo-mechanical action on sorghum fibre in the twin screw reactor is not sufficient to ensure a high quality to the raffinate. It is the chemical action which is predominant. The increase in sodium hydroxide content indeed favours the solubilisation of hemicelluloses^[23]. However, when sodium hydroxide is high, centrifugation of the matter is less efficient, and a higher proportion of the hemicelluloses is carried with the fraction. But also an increase in the sodium hydroxide concentration over 3.5 %, propitious to the solubilisation of a more important proportion of hemicelluloses, would make possible a better reorganisation of the fibrous matter under the action of the co-penetrating screws, leading to an improvement in the quality of the pulps. Thus, as it was suggested in many studies, hemicelluloses play a positive part in the manufacturing of paper pulp^[24,25]. In support of this assumption, the washing in water of the fraction which leads to a further extraction of 10 % of the initial dry matter, induces the weakening of the mechanical characteristics of the pulps (table 8), always except the tear index. The analysis of the unusual behaviour of this response must be counterbalanced by the fact that the second degree polynomial model we chose to establish a correlation with the temperature and sodium hydroxide content factors. Unlike the other responses, does not allow the presentation, in a satisfactory way, of the results corresponding to the examined field. This could point out that other factors interacting with the thermo-mechanical factors have a predominant influence on the tear index.

4. Conclusion

The fractionation of whole fibre sorghum in a twin-screw reactor is an interesting alternative to

mechanical depithing. The output achieved through mechanical depithing is of 50 %, which limits twin-screw paper pulp yield to 40 % of the initial sorghum. The pulp yield achieved by direct twin-screw fractionation is of 80 %. Even though the paper qualities of the pulps achieved by direct twin-screw fraction are slightly lower than those obtained from mechanically depithed sorghum fibre, they give entire satisfaction for an application in cardboard packaging.

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