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Application of Nano-Hydroxyapatite in Sugarcane Juice Clarification

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Abstract

In this study, the application of nano-hydroxyapatite (NHA) in the clarification of mixed cane juice "MJ" was investigated via batch experiments on a pilot scale. Control points (pH, doses, retention time, color, and turbidity) of this process were evaluated compared with the traditional sulfitation process. The results were evaluated using a statistical T-test. The results showed that the most proper pH, doses and retention time are 7.5, 300 ppm, and 1.5 hr, respectively. Since, applying such values led to a significant reduction in turbidity and color of clear juice (55 % and 19 % respectively) compared with the traditional sulfitation and phosphation process. These results mean a predicted soft scale formed in the evaporators, the lower color of produced sugar, higher sugar recovery, removing the cost of sulfur, and minimizing the cost of calcium oxide to half. The results were discussed via the properties of hydroxyapatite and its highest surface area that adsorbs denatured proteins, prevents the formation of melanoidin coloring compounds and promotes clarification efficiency.

Keywords:* *Hydroxyapatite nanoparticles; NHA; Clarification; mixed juice; Clear juice.

Introduction

The application of nanomaterials in the clarification processes in several industrial and agricultural sectors has attracted significant attention. Since it has a significant impact on the yield and quality of sugar produced (Madsen, 2009) Traditional clarification methods involves: Treatment with lime in water or syrup (saccharate) solution remains the basic method of clarification, normally termed defecation, where lime has been the universal chemical for neutralization of the acidic juice and the process varies in the method and temperature of addition. Variations of simple defecation have always the aim of lowering the color and turbidity of the clarified juice (Madsen, 2009). In addition to simple defecation there are three traditional methods known as sulfitation, phosphatation and carbonatation. Among of all, lime is used as a main clarification agent in the preliming step.

The main goal of clarification is to remove the coarse and colloidal materials without losing sugar. This goal must be achieved via several control points involves:

- Provide conditions of temperature, pH and ion concentration which will maximize the precipitation of soluble impurities from the juice .
- Flocs formation to trap all suspended matter which can be settled at a satisfactory rate (Doherty et al., 2002).
- Produce clarified juice of high quality, with a minimum turbidity, color and a low calcium (Ca^{++}) content and carry out the above at minimum cost, minimum residence time, minimum loss of sucrose, and minimum formation of color (Rien, 2007).

A variety of chemical and physical reactions occur during the clarification process, colour formation is concedered one of the most important among of these reactions where color is a complex mixture of compounds which stem from either the cane plant (e.g., chlorophylls, flavonoids and polyphenolic compounds) and/or produced during the processing of juice in the sugar factory for e.g., melanin's, melanoidins, caramels and aroma compounds (Mersad et al.., 2003; Nguyen & Doherty, 2011; Paton, 1992).

Colorants are a class of impurities in sugar cane juice that are difficult to remove and tend to occlude in sugar crystals (Paton, 1992) Changes in sugar cane juice pH and temperature contribute to the formation of colourants and colour precursors as a result of sucrose degradation and various other chemical reactions.

The colored compounds related to processing are classified into three main groups. (I) hexose alkaline degradation products (HADP) (II) melanoidins (Maillard reaction products), and (III) caramels.

All of these colorant classes are formed in the presence of reducing sugar, colour precursors (e.g., phenolic acids) are typically not removed during clarification and persist in the later stages of processing. These compounds can polymerize to high molecular weight polymers and react with metal ions in juice to form complexes which are associate with the color in raw sugar (Lindeman & Oshea, 2001). Flocculation of macro-flocs via particle bridging of calcium phosphate flocs impurities and copolymer flocculants, and adsorption of waxy-like materials and suspended solids with precipitate (Eggleston et al. 2002).

The aim of this work is to evaluate the most proper control points (pH, doses and retention time) in the presence of nano-hydroxyapatite, and its effects on turbidity and color behavior during clarification process for raw cane juice.

Materials and Methods

- **Preparation of hydroxyapatite nanoparticles**

Hydroxyapatite nanoparticles (NHA), $Ca_5(PO_4)_3(OH)$, were synthesized via wet-chemical precipitation Methods (**Salah *et al.* 2018)** and cited in previous paper, in press .

- Polarization of sugar juice was determined using polarimeter PolAAr 3001, England.
- Calcium was determined by titration with EDTA according to ICUMSA method GS8/2/3/4-9 (2000).
- Silica was determined as molybdosilicic acid using colorimetric method according to laboratory manual of ESIIC (1991).
- Batch settling test, initial settling rate, and final mud level were determined according to operating procedure for the SRI Settling Test Kit method, SRI (1999).
- Turbidity of the CJ was determined by measuring the absorbance (A) at 975 nm in 1 cm cuvette cells against Mille-Q water (Luoisiana).
- The brix of juice samples was measured at ambient temperature using a digital refractometer (PTR 46, Index Instruments Ltd., UK).
- The pH of juices was measured using a pH/mv/temp pH meter (Jenway 3510, England 027500).
- The% sucrose or Pol in cane juice is typically determined by measuring the positive rotation of polarized light via the double polarization method, using a PolAAr 3001 (Optical Activity Ltd., UK).
- Phosphate, was determined colorometrically using ammonium molybdate according to laboratory manual for South African Sugar Factories.
- Lime as milk of lime of baumme “± 6” obtained from sugar mill.

- Anionic flocculent used in experiments was Magnafloc $LT_{27,}$ Acrylamide/ acrylate co – polymer, Samoral, MW18 x 10^6, and degree of hydrolysis 27.

Flocculant solutions of 0.1 % (w/v) are more viscous and the best preparation method (Madsen 2009) was to disperse and dissolve the flocculent powder in deionized water by gentle stirring using a small propeller stirrer at a low shear rate of 50 rpm for 3 hr.

Clarification procedure in presence of (NHA).

NHA clarification process has performed according to the following 30 L of MJ delivered to reaction tank, sample of 500 ml mixed juice was taken for analysis. " NHA " added as asuspension with 300 ppm dosage. Milk of lime "$Ca(OH)_2$ added to pH 7.5 at 75 ^oC, boiling at 103 ^oC. Allowing flashing of juice and settling in the clarifier through retention tim 1.5 h. Sample of 500 mill clarified Juice collected as a sample for evaluating the efficiency of ” NHA” clarifying agent. In each test the evaluation based on the comparison between the control points" brix, purity, Ca % brix, P_2O_5 ppm, SiO_2 ppm, turbidity and colour of clarified juice resulted from traditional and nano clarification.

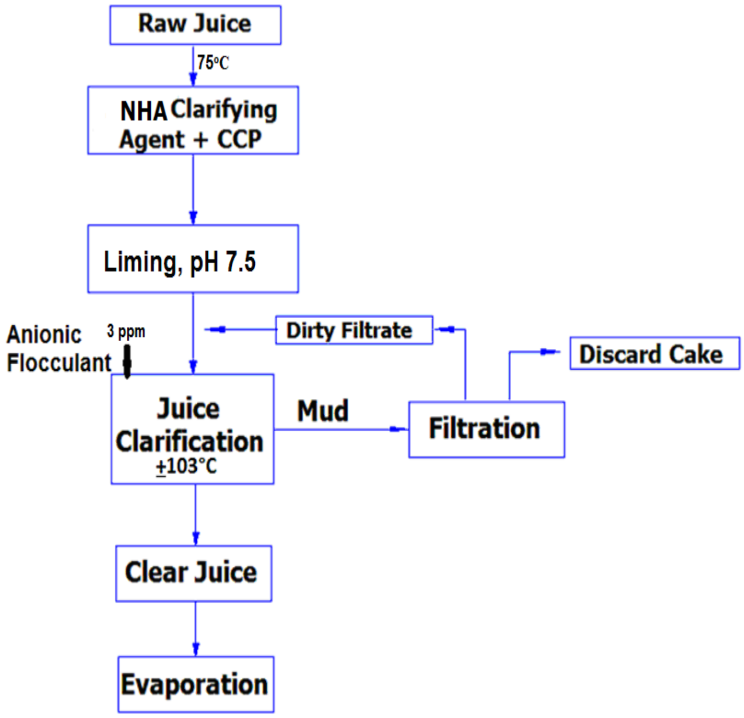

Fig. 1: Clarification process flow sheet in presence of "NHA"

Sulfitation process

Sulfitation clarification process in factory was followed according to the following flow sheet (Fig. 3)

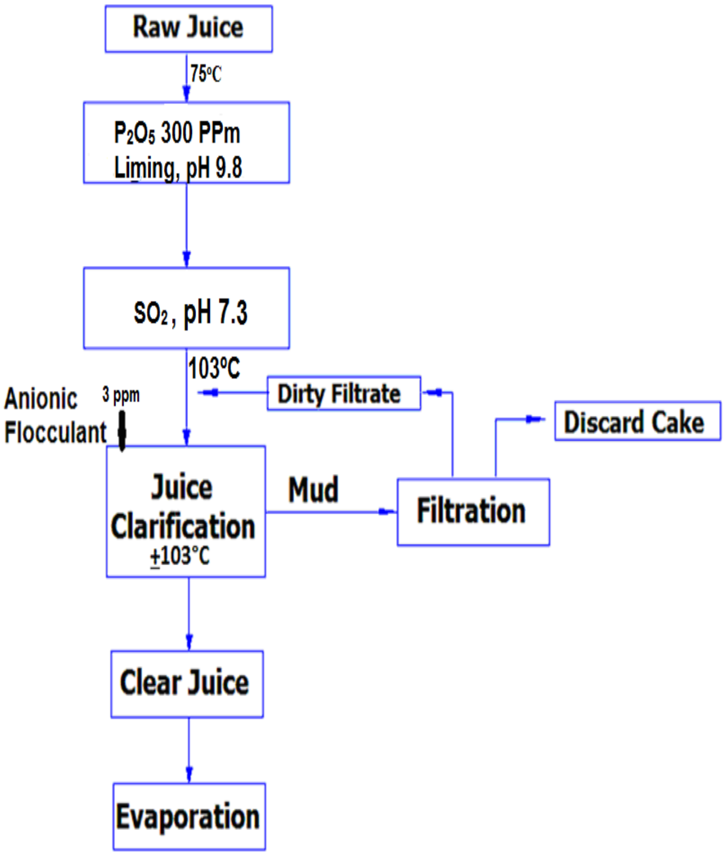

Fig. 2: Flow sheet of sulfitation process

Statistical analysis

Using Microsoft" Office Excel (2007), Statistical differences between two sets of data assuming equal variances were determined at the 5% probability level.

Results and Discussion

- **Samples for compositional analysis**

Factory mixed juice samples from Geurga Sugar factory "represent the milling tandem process and Quos sugar factory "represent diffuser moving bed in the extraction of juice. The decade analysis of mixed juice from the stand point of physic chemical properties" brix, pol % brix, purity, reducing sugar, pH, and ash % brix were analyzed (Table 1).

Table 1 : Average composition of cane juice extract by milling and diffusor

Year extractor	Pol in % juice g /100 g	Reducing sugar g/ 100g	Purity	Ash %Bx	pH
2015					
Mill *	**13.06**	**5.63**	**82.83**	**4. 51**	**5.6**
	± 0.01	**± 0.02**	**± 1.2**	**± 0.2**	**± 0.06**
Diffuser*	**13.58**	**5.52**	**84.33**	**3.58**	**5.4**
	± 0.02	**± 0.02**	**± 1.5**	**± 0.15**	**± 0.07**
2016					
Mill *	**12.13**	**5.92**	**80.8**	**4.35**	**5.7**
	± 0.02	**± 0.02**	**± 1.3**	**± 0.1**	**± 0.07**
Diffuser*	**12.58**	**5.36**	**81.26**	**3.74**	**5.3**
	± 0.01	**± 0.01**	**± 1.1**	**± 0.12**	**± 0.06**

***Diffusor in Quos sugar factory & milling in George Sugar Factory**

Dosage test

Using different dosages (200,300 and 500) of NHA clarifying agent, clarification were conducted (figure 1), for determining which dosage gives the best clarification performance in terms of juice turbidity, final mud level, initial settling rate of flocs, colour and levels of inorganic ion for untreated MJ & clarified juice.

Table 2: Results of mixed juice (MJ) clarification at different dosage of "NHA" Process

Sample No.	Mixed juice(MJ).	Clarified juice(CJ)		
		200 ppm	300 ppm	500 ppm
Initial settling rate C.M /min	-	22	30	29
Settling behavior	-	Moderate	Fast	Fast
Final mud level(%)	-	20% * ± 2 %	10%* ± 1	12% ± 2
Turbidity	-	24*	12*	14
Ca (mg/kg	0.66 ± 0.03	0.74 ± 0.04	0.71 ± 0.02	0.74 ± 0.03
P (mg/kg on Bx	160 ± 6	22 ± 3	18 ± 2	20 ± 4
Si (mg/ kg on Bx	238 ± 13	168* ± 10	119* ± 9	125 ± 7
Juice pH	5.4 ± 0.1	6.7 ± 0.1	6.7 ± 0.1	6.6 ± 0.1
Purity.	83.8 ± 0.3	83.4 ± 0.2	83.6 ± 0.1	83.6 ± 0.2
Color	-	10010 ± 170	8903 ± 150	8703* ± 200

***Correlation are significant at $p < 0.05$**

Table 2 shows the results related to dosages tests, in which the effect of changed doses up on settling rate behavior, turbidity and the level of inorganic ions were followed, 300 ppm added dosage from " NHA " clarifying agent is the most proper one, since it resulted in the lowest final mud level (12 %) and the lowest Ca^{++} and Si^{+++} level (0.71% and 119 ppm respectively) in the clear juice.

Relatively high (CJ), Turbidity results in each case, inspite of fast settling behavior with dosages 300 and 500 this may due to the small flocs which attributable to high impurity load of (MJ) and the inability of the flocculant to effect bridging of the calcium phosphate floes , thus using of cationic flocculant before addition of NHA clarifying agent will facilitate the aggregation of colorant to be adsorbed on the surface of nano-hydroxyapatite.

- The color of clear juice resulted from this technique also can be improved by using cationic flocculent before addition of "NHA", where the dosages 200, 300 ppm resulted adaptively moderate (CJ) color "8903 and 8703 respectively ".
- A good advantages for HAP clarification is the resulted moderate P_2O_5 level in the (CJ) at each dosage, (20, 18 and 20 respectively).

pH" s Degree

Firstly, automation control pH's must be followed to avoids the troubles in clarification and hence dissociation of calcium phosphate formed, changes in the overall charges upon the serves aria which affect the clarification processes.

Since cane juice is acidic, corrosion of mild steel components in the diffuser is an important consideration. Liming in the diffuser is thus practiced to control pH. It is possible to control corrosion by raising the pH to values between 5.5 and 6.0 conventionally with milk of lime.

Table 3: Results of (CJ) from HAP clarification at different pH

Items	M.J	pH 7	pH 7.5	pH 8
Brix	**13.6**	**13.2**	**13.3**	**13.3**
	± 0.01	**± 0.02**	**± 0.02**	**± 0.02**
Purity	**83.4**	**83.0**	**83.2**	**83.0**
	± 0.9	**± 0.8**	**± 0.7**	**± 0.8**
Turbidity	**-**	**11**	**7**	**12**
		± 2	**± 1**	**± 2**
Color	**-**	**9790**	**9240**	**10713**
		± 120	**± 72**	**± 90**
Ash	**3.2**	**3.1**	**2.9**	**3.0**
	± 0.1	**± 0.1**	**± 0.1**	**± 0.2**
Ca^{++}	**0.66**	**0.73**	**0.71**	**0.76**
	± 0.02	**± 0.03**	**± 0.02**	**± 0.03**
P_2O_5	**160**	**22**	**12**	**28**
	± 11	**± 3**	**± 3**	**± 3**
Si	**240**	**151**	**110**	**142**
	± 15	**± 12**	**± 9**	**± 11**

Results of (CJ) from clarification at different pH (Table3) show that the most proper pH degree at (7.5). Since the lowered color of produced clear juice (9240), and the lowered Si, CaO content, and turbidity in CJ (110 ppm, 0.71 % Bx and 7 NTU) respectively. This points must be taken as a pH control point for “HAP” clarification

For understanding the effect of pH we must be undertake the effect of liming technique in our consideration However, high pH values and/or overliming must be avoided for the following reasons:

- pH values above 7 cause percolation problems by affecting the nature of the fiber irreversibly Percolation velocity is reduced considerably (Love and Rein, 1980).
- Hydrolysis of the acetyl group in the hemicellulose fraction of the cane fibre can occur, This produces calcium acetate which in turn produces acetic acid, The acid is volatilised in the evaporators causing very severe corrosion in calandrias, and in vapour and condensate piping. Laboratory

work has shown that extraction of silica, found in relatively high concentrations in tops and trash, from cane is increased as the pH rises (Walthew et al, 1988). Silica has been associated with scaling problems in the evaporators. Laboratory work has shown that high pH values favour the extraction of colour bodies, particularly from tops and trash (Lionnet, 1985). Good pH control is essential, but it is not easy.

Retention time

The results obtained from clarification of MJ in presence of " NHA" at different retention time (RT) show that, 1½ hr is the most proper retention time. Since, a lower turbidity in clear juice (7 NTU), low CaO % sugar in clear juice (0.61%), moderate P_2O_5 in ppm % brix in clear juice (13 ppm), and the color of resulted clear juice (8831) in the rang of producing white consumption sugar.

Table 4 : Results of clarification of (MJ) in presence of "NHA" at different (Rt)

Items	MJ	CJ at different Rt		
		1 hr	1½ hr	2 hr
Purity	83.4 ± 0.4	83.2 ± 0.3	82.9 ± 0.1	82.6 ± 0.2
pH	5.4 ± 0.1	6.7 ± 0.1	6.7 ± 0.1	6.6 ± 0.1
Ca	0.62 ± 0.04	0.64 ± 0.3	0.64 ± 0.03	0.68 ± 0.02
Si	234 ± 12	112 ±3	113 ±2	119 ± 1
Turbidity		15 ± 1	7 ± 3	9 ± 1
P_2O_5	162 ± 3	14 ± 2	11 ±1	15 ± 1
Color mau		8940 ± 140	8831 ± 70	9710 ±200

The effect of retention time on color, turbidity, and sugar recovery in the boiling house was investigated and established via previous studies ,The retention time in the clarifier during sulfitation process equal "1.5" hr. so it is an advantage point to equal "1.5" hr in this process, this means that there is no change in the design of clarifier during application of " NHA" in can juice clarification.

Effect of NHA on turbidity

Turbidities of CJ (Table 5 and Fig. 3) obtained for both sulfitation process and clarification in the presence of HAP. The average turbidities in the case of "NHA" clarification (4) Lowerthan that obtained with sulfitation (9).The results show that "NHA" clarification process was more effective method in reducing CJ turbidity.

Table 5: CJ turbidities units during " NHA" clarification and sulfitation processes.

Sample No.	Clear juice from	
	Sulfitation	**NHA clarification**
1	**9**	**2**
	± 1	**± 1**
2	**12**	**5**
	± 2	**± 1**
3	**7**	**4**
	± 1	**± 2**
4	**11**	**6**
	± 1	**± 1**
5	**6**	**3**
	± 2	**± 1**
6	**9**	**5**
	± 1	**± 2**

These results may explanted due to the properties of NHA as follows:

- More open floc structures in the case of "NHA" clarification. Loosely bound and highly branched flocs are attributable to effective trapping of micro-flocs, and adsorption of particle impurities onto the surface of the floc network (Salah et. al. 2014)
- In the case of using nano-hydroxyapatite, calcium phosphate has $Ca_5(PO_4)_3(OH)$ form. this different morphological structure appeared in the form of thin short nanorods, exhibits excellent adsorption ability

because its large specific surface area, and thus an additional improvement in the performance of clarification process.

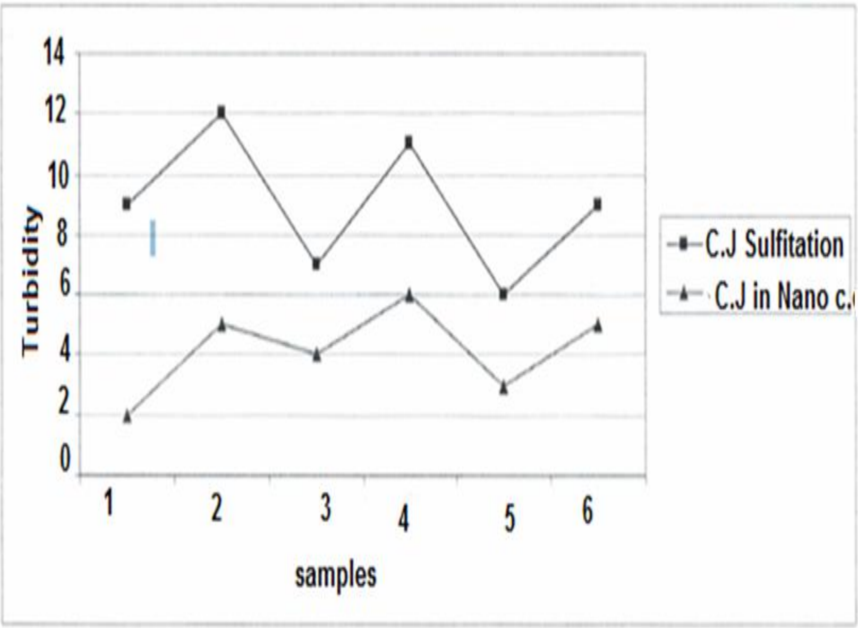

Fig. 3: CJ turbidities units during sulfitation and " NHA" clarification

Behavior of colour

As shown from Table (6) a significant different in color of CJ resulted from NHA clarification. The colour of C.J resulted from clarification using NHA increase by about (8 %). more than that in the case of sulfitation. Where as the colour of C.J resulted from traditional phosphtation increase by about 27 % more than that resulted from sulfitat , this means that using of HAP leads to lower the colour of C.J by about 19 % compared with phosphtation processe

Table 6: Behavior of color in case of nano technology and sulfitation**.**

Replicate	Sulfitation	NHA clarification	Phosphtation
1	8345 ±70	9256 ± 20	10890 ± 90
2	8444 ± 81	8975 ± 32	10685 ± 87
3	8300 ± 72	9000 ±41	10834 ± 102
4	8250 ± 70	8900 ± 52	10117 ± 92
5	8345 ± 72	8953 ± 38	10411 ± 82
Av. % increase	—	8%	27 %

These results may explained via, nature of colorant and properties of NHA, where the colored compounds related to processing are classified into three main groups (I) hexose alkaline degradation products (HADP) (II) Melanoidins (Maillard reaction products), and (III) caramels. All of these colorant classes are formed in the presence of reducing sugar, colour precursors (e.g., phenolic acids) are typically not removed during clarification and persist in the later stages of processing. These compounds can polymerize to high molecular weight polymers and react with metal ions in juice to form complexes which are associate with the color in raw sugar (Lindeman & Oshea, 2001).

"Melanoidins" are colored molecules that are produced when a reducing Sugar is heated in the presence of amino compounds, specifically amino acids.

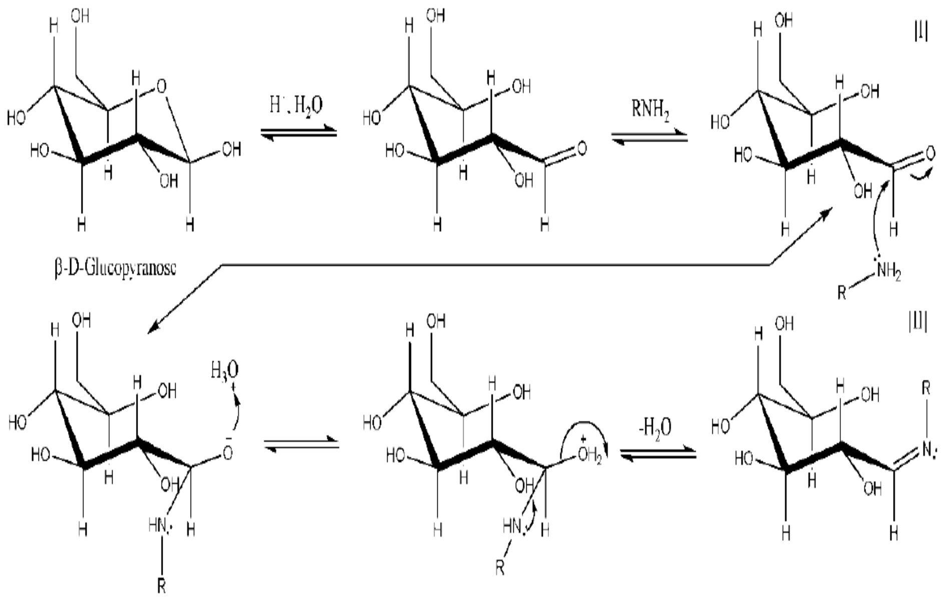

Fig. 4: Amadori rearrangement of D-glucose to D-fructose via Schiff base.

In addition to caramelization, if amino acids, amines, or NH_3 are present, a cascade of reactions known collectively as the "Maillard reaction" can occur with reducing sugar to yield dark brown insoluble polymer, frequently in excess of 20 kDa (Godshall, et al., 1987; Lindeman, 2001). The "reaction" is general in that reducing sugars will react with practically any amine to yield a multitude of products.The amine reacts with the open chain carbohydrate to yield an unstable intermediate which dehydrates to yield the corresponding "Schiff base" or imine (Hodge, 1953a).

In the case of using nano-hydroxyapatite instead of traditional tricalcium phosphate, the effectiveness of the juice color removal would be considerably increased. This due to its morphological structure, which appeared in the form of thin short nanorods, exhibits excellent adsorption ability because its large specific surface area, and thus an additional improvement in the color removal during NHA clarification process (**Salah *et al.* 2014).**

It has been established that, the challenge is to develop conditions during clarification conductive agood methed instead of traditional sulfitation methed for high proportion of HAP (Doherty and Rackeman 2009).

Cost evolution of the HAP clarification for mixed cane juice

We assume a cane sugar factory processing 1500000 tons per crushing season. according to our experiment, for comparison between sulfitation and NHA clarification process from the stand point of cost evaluation, Firstly the chemical aid and its consumption in each case were determined (table 7and 8) the cost and the benefit were evaluated (Table 9 and 10) according to the quantity of chemical consumed in each case.

Table7: Chemical consumption % cane in case of sulfitation & NHA clarification

Chemical aid	**Consumption in**	
	Sulfitation	**NHA clarification**
CaO % cane	**0.16 % cane**	**0.08% cane**
$H_3 Po_4$	**0.03% can**	**-**
Hydroxy apatite (NHA)	**-**	**0.03% cane**
Anionic fluctuant	**0.0004% can**	**0.0004% cane**
Sulfur	**0.05% cane**	**-**

Table 8: Quantity of chemical consumption per year in case of sulfitation and NHA clarification

Chemical aid	Quantity of chemical consumed	
	Sulfitation	NHA clarification
Calcium oxide	2400 ton	1200 ton
Ortho phosphoric acid	450 ton	-
Sulfur	750 ton	-
Anionic fluctuant	7.5 ton	7.5 ton
NHA	-	450 ton

Cost of chemical consumption in case of sulfitation and HAP clarification in million L.E were evaluated Table. 9

Table 9: Cost of chemicals in case of NHA & Sulfitation- clarification

Chemical aid	Cost	
	Sulfitation	HAP clarification
Calcium oxide	3.154.000 L.E	1.580.000 L.E
Ortho phosphoric acid	3.710.000 L.E	-
Sulfur	2.500.000 L.E	-
NHA	-	5.400.000 LE

Table 10: Total cost of NHA and the total benefits .

Cost	Benefit
NHA = 5.400.000 L.E	Removing of sulfer cost = 2.500.000
	minimizing of Cao cost to the half 1.580.000
	removing cost of phosphoric acid = 3.710.000 L.E
	In addition to maximize the recovery of crystallization pans, not calculated
	decreasing losses in final molasses not calculated
Total cost 5.400.000 L.E	Total Benefit 7.790.000 L.E

From the stand point of economic study the cost to benefit ratio 1:1.44, i.e the net profit = 2.390.000 L.E per year, take in mind that the increase in sugar recovering not calculated. for all factories related to Esiic the net profits per year = 14.34 m. L.E .

Conclusion

The work conducted in this paper has shown that the control points for applications of NHA as nano clarification in cane sugar industry in term of pH, doses, and retention time, were determined as 7.5, 300 ppm, and 1.5 hr respectively to be the most proper under conditions of Egyptian cane juice. Since, under such parameters, the clarification of mixed juice using nano hydroxy apatite NHA can be used as a good method instead of traditional sulfitation method, since it leads to brilliant CJ with a lower turbidity (4.n.t.u) and color (7166 mau), lower Ca.% sugar in CJ (0.6 %), a moderate P_2O_5 ppm (16 ppm) in CJ, and a lower SiO_2 ppm % BX (105) in CJ. These results mean a predicted soft scale formed in the evaporators, overcoming the hard to boiled massecuite phenomenon, removing the cost of sulfur, and minimize the cost of calcium oxid to the half

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الملخص العربي

تطبيق استخدام النانو هيدروكسي ابيتايت في معالجة عصير قصب السكر

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[2]معهد بحوث ودراسات تكنولوجيا صناعة السكر - جامعة أسيوط
[3]وحدة بحوث و تطوير العمليات الصناعية - شركة السكر والصناعات التكاملية المصرية

تم فى هذا البحث دراسة تطبيق استخدام مادة النانو هيدروكسي ابيتايت و تقييم استخدامه كعامل معالجة ضمن عملية معالجة العصير الخليط لصناعة السكر .

ويعتبر الهدف الرئيسي من هذه الدراسة هو توصيف النقاط الحاكمة (pH ، العكارة ، اللون ، زمن الاحتجاز ،الجرعات المضافة جزء/ مليون) فى حالة تطبيق هذه الطريقة كطريقة معالجة لتقيمها مقارنة بطريقة الكبرتة العادية المتبعة بالمصانع من خلال التجارب التى تم اجراؤها على نطاق معملى ونطاق نصف صناعى .وضحت النتائج بعد إجراء التحليل الاحصائى باستخدام اختبار T أن درجة أس ايدروجينى 7.5 ، زمن احتجاز 1½ساعة ، جرعة 300 جزء/ مليون جزء عصير ،هى أفضل ظروف لعملية المعالجة باستخدام النانو هيدروكسى ابيتايت ، حيث أدى تطبيق هذه الظروف على معالجة العصير الى انخفاض واضح فى درجة العكارة وصل الى 55% وانخفاض فى اللون وصل الى 19 % عن طريقة الفسفتة العادية. وهذه النتائج التى تم الحصول عليها بعد اضافة مركب النانو هيدروكسى ابيتايت ضمن عملية المعالجة ، تعنى إمكانية الحصول على رواسب أقل كماً وأقل صلابة فى مرحلة التبخير ومن ثم ارتفاع فى معدل الانتقال الحراري ، كما تعنى التغلب على ظاهرة صعوبة البلورة للماسكويت نظراً لانخفاض الكالسيوم والسليكا بالعصير الناتج بالإضافة الى توقع انخفاض لون السكر المنتج وزيادة الاستخلاص وانخفاض فقد السكر بالمولاس.

وقد تم تفسير النتائج السابق ذكرها اعتماداً على خواص مركبات فوسفات الكالسيوم وميكانيكية الارتباط والتجميع للغرويات العالقة والتى تعتمد على مساحة سطح المواد المضافة للمعالجة والتى تصل أعلاها فى حالة استخدام النانو هيدروكسي ابتيايت الذى يمتز البروتينات الغروية فيمنع تكون الميلانودينات الملونة ويؤدى الى خفض درجة عكارة ولون العصير الرائق الناتج وزيادة كفاءة آداء عملية المعالجة للعصير بوجه عام .