Investigation into The Filterability of Raw Sugars from Different Geographical Regions of The World

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ABTRACT

This study was carried out at Al khaleej Sugar Refinery in Dubai, United Arab Emirates. The study aimed to find out the correlation between the filterability and slurry resistance values of raw melt liquor in the carbonation sugar refinery and the raw sugar quality.

The investigation utilized 5 raw sugar samples collected from various regions of the world comprising three quality groups namely; Low Pol sugar (LP) from Thailand, Very High Pol sugar (VHP) from Brazil , South-Africa , Sudan , India , and Very Very High Pol sugar (VVHP) from Brazil.

The filterability and the slurry resistance tests were used as the main determining factors for the evaluation. The filterability of refined sugar (considered as 100 %) was used as a reference value for comparison.

The results of the experiments showed that the filterability of (L P) raw sugar was only about 20% from that of refined sugar. Whereas the filterability values for (VHP) and (VVHP) sugars ranged between 40% to 80% from that of refined sugar.

In addition, the slurry resistance values for VHP and VVHP sugars in the laboratory were 0.76 and 0.64 respectively compared to 0.74 and 0.48 in the refinery production line.
It was also observed that there is a close similarity between laboratory filterability and slurry resistance with the actual refinery filtration process which suggests that the laboratory filterability and slurry resistance tests could be used as tool to predict the behavior of the refinery filtration process for similar qualities of raw sugar.

INTRODUCTION

Filteration process is an old well-established unit operation which is traced back to 1789. It is used in sugar refining industry to remove the suspended nonsugar particles from the melted raw liquor after the purification stage (Orr,1977).
Clarke (2000), stated that the knowledge of the composition of raw sugar is essential in order to optimize the refinery operations and the selection of suitable methods of purification for the efficient removal of nonsugar components.
Raw melt liquor purification can be affected either by carbonation process or phosphotation process . During these processes most of the impurities are precipitated either in the form of calcium carbonate from carbonation refinery or calcium phosphate from phosphatation refinery.
The nonsugar content of raw sugar has been studied by many research workers, Siato et al(1976) and Chou(1991). They stated that the nonsucrose content of the raw sugar is different from one country to another, qualitatively and quantitatively.
The efficiency of each stage of the refining process is influenced in different ways by the quality of raw sugar and depending on the type of refinery, its clarification and decolorization processes, some components are easy to remove, and others are more difficult.

Raw sugars from various regions of the world filter differently ; though they might be from the same quality brand .Some filter well and others give poor filterability .The reasons are normally

Vianna (2000) stated that no single factor that would impact on raw sugar filterability, but rather a combination of several factors. However, the parameters correlating most strongly with poor filterability correlated to the specific quality characteristics of the sugar.

Filterability is defined as the ability of raw sugar to filter easily. It is an indication of the filtering characteristics of a raw sugar when it is processed through the clarification stage of the refinery.

are: turbidity, soluble phosphate, starch, suspended solids, and dextran.

There are three major brands of the raw sugar; Low Polarization abbreviated as (LP) with Pol < 99 % , Very High Polarization (VHP) with Pol 99.3 %, and Very Very High Polarization (VVHP)with Pol about 99.65 % (Rein,2007).

Raw sugars with poor filterability will be directly reflected in the refinery capacity .The liquor flowrates will be reduced due to difficulty in filtration . Hence , the operating personnel will find themselves forced to choose one of the following remedies:

1. Reduce the liquor brix which will lead to more consumption of steam at the evaporators to remove the water.
2. Increase the temperature of the liquor in order to reduce the viscosity , hence boosting the flowrates .
3. Add more alpha amylase enzyme when raw sugar is having high level of starch .
4. Increase lime ratio to give better purification performance and act as filter aid for filteration.

5. Add additional filter aid to reduce the insoluble solids and the turbidity levels of the filtrate.

There are two main parameters which are used as indicators for the evaluation of raw sugar filterability:

1.The (%) filterability.

2.The slurry resistance .

Both indicators are very important criteria to be used for predicting the behaviour of raw melt liquor at the filteration station of the sugar refinery .

Filterability (%) is defined as : the cumulative volumetric flow rate (ml) of filtrate collected from filtering raw melt liquor at 65 Brix and 8.2 pH at 80C - 85C temperature through a precoated filter paper of (8 microns) in 10 minutes ; divided by the flowrate of a pure refined sugar solution with similar conditions Winn(1979)

$$(\%)\ \text{filterability} = \frac{\text{ml of raw melt liquor @ 65 Brix}}{\text{ml of refined sugar solution @ 65 Brix}} \times 100 \quad \text{..eqn 1}$$

The slurry resistance of the formed cake was calculated using the following formula Field (2001) :

$$\text{Slurry Resistance (S.R)} = \frac{\text{Slope of linear line} \times 9.8 \times 10^{5}}{\text{Brix}^{2} \times \text{density}^{2}} \quad \text{....eqn 2}$$

A graph of (filtration time of treated raw melt liquor divided by accumulated volume of filtrate versus accumulated volume of filtrate during that time) was plotted for each sugar sample and the slope of the linear line was calculated, then the slurry resistance value. The filtration time in this test was 10 minutes.

MATERIALS AND METHODS

Chemical Reagents:

The chemical reagents used in the experiments were prepared according to the International Commission of Uniform Methods of sugar analysis (ICUMSA) methods book # 4 published in (1994) and supplemented in (1998).

Distilled water with a conductivity ash less than 2µs/cm was used as the solvent for preparation of sugar solutions, and for preparing chemical reagents. The reagents used in the experiments of filterability determination included:

1. Buffer solution: pH9.
2. Milk of lime solution: of 15 degree Baume.
3. Filter aid: Celite-505, Hyflosupercel.
4. Filter paper: 8 microns, and 0.45 micron filter papers.

Apparatus:

The apparatus and devices used for the laboratory experiments included:

1. Volumetric Glassware & Plastic bottles: various volumetric glassware and plastic bottles with special 100 ml flasks.
2. Water Bath: from Memmert Co., model WB-14, with mounted control thermostat, capable of controlling temperatures between: (10ºC to 100ºC) and with precision of ± 0.03C.
3. Thermometers: with the readable range from (-140 to 110ºC) and with precision of ±0.1ºC.
4. Refractometer: from Atago Co., model RX-5000, digital, with the range from (0.0-100) and precision of ±0.03%.

5. pH Meter: from Hanna Instruments, model HI-9321, a microprocessor pH meter, with the range from (0-14 pH) and the precision of ±0.01 pH.
6. Filtration Apparatus: from Millipore Co., comprising a conical filtration flask of capacity 1 liter, holder for membrane filter fitted into filter connected with a vacuum pump which can read upto 625 mmHg, and Buchner Funnels.
7. Filterability equipment: a jacketed filtration unit equipped with CO_2 gas bubbler mounted on the bottom of reaction vessel and a measuring cylinder.
8. Membrane filters: diameter 50 mm, pore size: 8 micron, 0.45 micron.
9. Membrane filter holder: fitted with SS support.
10. Measuring cylinder: to measure upto 200 ml.
11. Shaker and magnetic stirrers.
12. Stop watch.

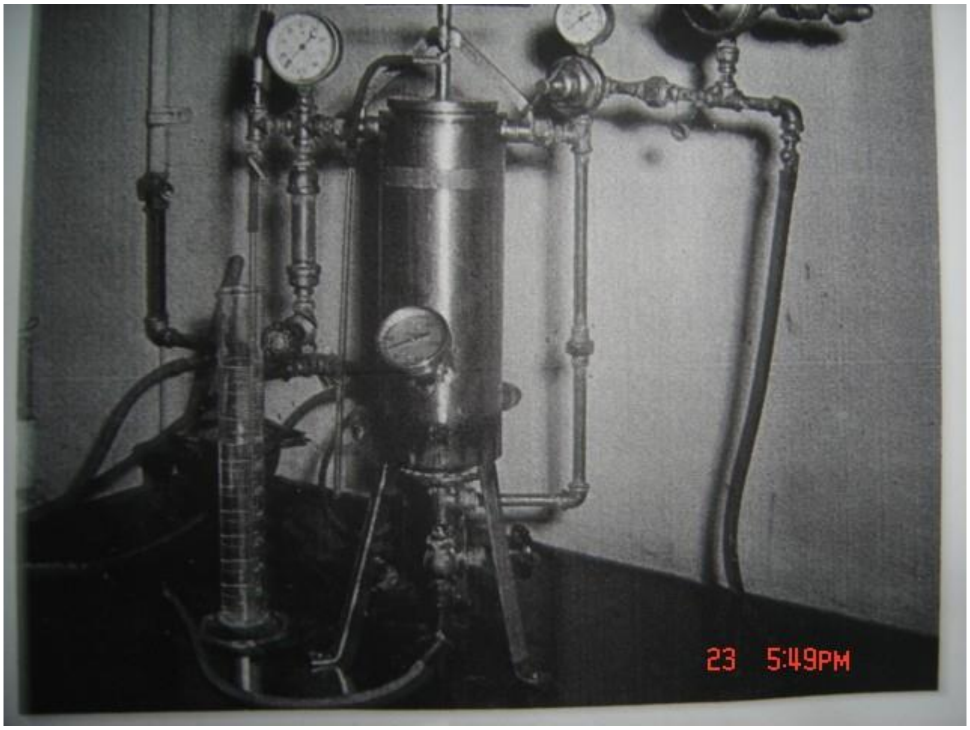

Figure 1: Laboratory Filterability Determination Equipment

Source:Al khaleej Refinery Laboratory

Methods of analysis

Determination of the Filterability of Raw Sugar Samples using Laboratory Filtration Equipment:

This method is the modified Australian Nicholson Horsely filtration determination method (Lab. Manual for Australian Sugar Mills, 1984). It is based on the comparison between the volume of filtrate of the test solutions of various raw sugars with the volume of filtrate of a pure white sugar solution under standard and identical conditions.

The calibration test performed using pure refined sugar was firstly done, and the volume of the filtrate was collected after 5 minutes and used as the reference to calculate the filterability of different test samples.

Procedure:

A sample solution for each raw sugar was prepared and adjusted to 65.0 brix in a Plastic Bottle. The pH of the solution was adjusted to pH 8.2 using buffer solution pH 9. The filter aid Celite Hyflosupercel was added (0.5% on solids). The mixture was stoppered and heated in a water bath to 80ºC with gentle shaking. The filtration apparatus was assembled and mounted in a water jacket. Water was circulated in the jacket and maintained at 80ºC. The sample was transferred to the filter funnel having 8 micron filter paper and the stop watch was simultaneously started. The filtrate volume for each sample was collected after 5 minutes in a measuring cylinder for the calculation of filterability. The accumulated filtrate volumes against filtration time was recorded for 10 minutes. Finally a graph was plotted using the accumulated filtrate volume versus the filtration time.

For standardization, a pure refined sugar solution was prepared and adjusted to 65 brix, and the same procedure for filtering raw sugar samples was followed.

The volume of filtrate collected after 5 minutes was recorded. The filterability of each raw sugar sample was calculated against the refined sugar volume.

Calculations:

The filterability of each raw sugar sample was calculated using the equation:

$$\% \text{ Filterability} = \frac{\text{Vol of filtrate of the test solution}}{\text{Vol of filtrate of the pure sugar solution}} \times 100 \quad \text{..eqn 3}$$

Determination of Slurry Resistance for the Carbonated Liquor from Raw Sugar Samples in the Laboratory:

This method depends on performing a batch carbonation process in the laboratory using laboratory filterability determination equipment, (Lee and Donovan, 1996). Figure 1. It is used to determine the slurry resistance of carbonated liquor for any type of raw sugar.

Procedure:

A solution of each raw sugar sample was prepared and adjusted to 65.0 brix in a 4 liters conical flaks. The pH of the solution was adjusted to 10.2 using milk of lime. The temperature of the solution was maintained at 80ºC in a thermostatically controlled water bath. CO_2 gas was bubbled into the conical flask. The solution was kept in a gentle stirring till the final pH was 8.2. Then the filterability and slurry resistance of the formed carbonated liquor were obtained from the graph plotted between the accumulated filtrate volume versus the ratio of filtration time / accumulated volume.

The slurry resistance was calculated using the formula by (Field, 2001):

$$\text{Slurry Reistance (S.R)} = \frac{\text{Slope of linear line} \times 9.8 \times 10^5}{\text{Brix}^{\wedge}2 \times \text{density}^{\wedge}2} \quad \text{.....eqn 4}$$

Determination of the Filterability of Actual Refinery Carbonated Liquor Samples from VHP and VVHP Raw Sugars:

For the determination of the filterability of actual refinery liquor, 50 ml of the carbonated liquor samples were collected from the carbonated liquor tank just before the filtration process on 2- hourly basis. Those samples were composited every 24 hours, then heated to 80ºC . The brix and pH were measured . Finally the sample was analyzed for the filterability determination using similar procedure as for the raw sugar samples (Laboratory Manual for Australian Sugar Mills, 1984).

This procedure was followed with two different raw sugars namely, VHP and VVHP. Analysis for the filterability were tripled for each type of raw sugar and the test results were averaged.

The four raw sugar samples were collected from traceability store of Al Khaleej Sugar Refinery laboratory. Those samples were from raw sugar ships received from; Brazil, India, Thailand, and South-africa. Also one sample from Kenana Sugar Company-Sudan collected from the crop/season: 2007/2008.

Those samples were given designations as below:

1.sugar from Thailand was designated as (A).

2. sugar from Brazil was designated as (B).

3. sugar from South-Africa was designated as (C).

4. sugar from India was designated as (D).

5. sugar from Sudan was designated as (E).

Treated melt liquors from all sugar samples were subjected to filterability test determination using laboratory filtration unit. The results of the determinations were presented in Table1, and fig. 2.

Further , the slurry resistance of each treated liquor sample was calculated using equation 2. The results were presented in Table 2. The graphical representation was shown in Fig. (3 - 7) inculsive.

RESULTS

Table 1 ,shows the results of filterability determination for all five raw sugar samples .

Sugar (A) is a low polarization sugar . It gave the least volume of filterate in 10 minutes ; which is a clear evidence for being of poor filterability at the filtration station of the refinery .

Sugars (B,C,D,and E) are very high polarization sugars .By comparison, sugar-B gave the highest volume of filtrate in 10 minutes , which is an indication for very good filterability at the filtration station of the refinery. Whereas, Sugar-D gave the least volume of filterate in 10 minutes ; indicating that it is of poor filteration behaviour among the same quality brand of sugars .

Table (1) : Results of Filterability test (ml /10 mins) for the five Raw Sugar Samples under Investigation

Time		*Raw*	*Sugar*	*Samples*	
(Mins)	*A*	*B*	*C*	*D*	*E*
1	10	28	24	12	16
2	13	47	34	17	30
3	16	54	41	21	38
4	18	60	45	25	45
5	20	65	48	28	52
6	22	68	50	30	57
7	23	71	52	32	60

8	*24*	*73*	*54*	*33*	*63*
9	*25*	*75*	*55*	*33*	*65*
10	*25*	*76*	*56*	*34*	*67*

The graphical representation for the filterability of all the sugar samples under investigation is demonstrated in Figure 2 below.

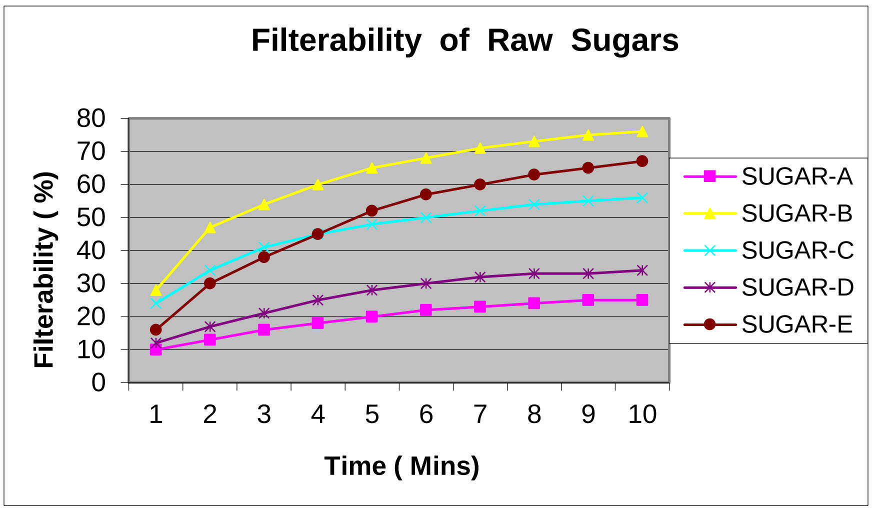

Figure (2) : Filterability of raw sugar samples from various regions of the world .

The slurry resistance values of the five raw sugar samples under investigation were calculated and reported in Table 2.

It is clear Table 2 that sugar–A gave the highest slurry resistance value, whereas sugar–E gave the lowest slurry resistance value .

Comparing sugars (B,C,D and E) which are of the same quality group of VHP, sugar E unexpectedly gave the lowest slurry resistance value ; in other words, the best filterable sugar . However, sugar-B was the one gave the highest filterability. This result showed some contradiction between the filterability and the slurry resistance value .The possible reason could be due to the starch content of sugar-E which is the lowest among all the VHP sugars .Starch has a drastic impact on calcium carbonate crystals which affects filtration.

Table(2): Slurry Resistance Values for Raw Sugar Samples

Sugar sample	Slurry resistance	Remarks
A	7.76	***The worst filterable***
B	0.82	
C	1.83	
D	3.53	
E	0.70	***The best filterable***

Figures from 3 to 7 are graphical representation of the slurry resistance values of the five sugars under investigation .

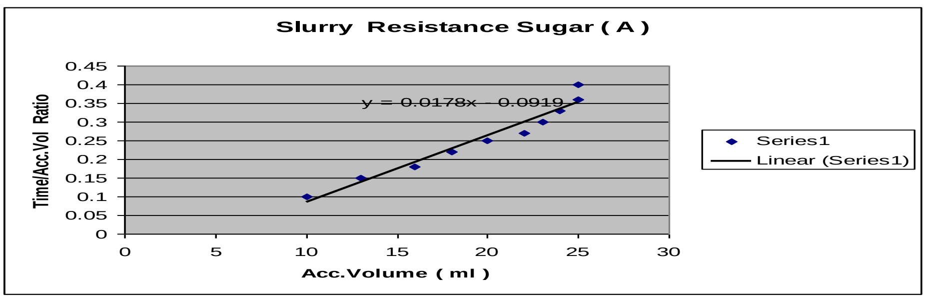

Figure (3) : Slurry resistance of Sugar (A **)**

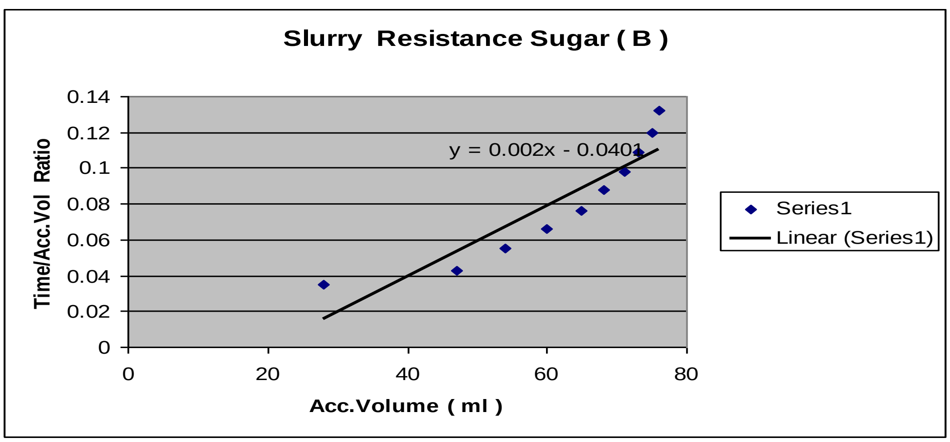

Figure (4) : Slurry resistance of Sugar (B)

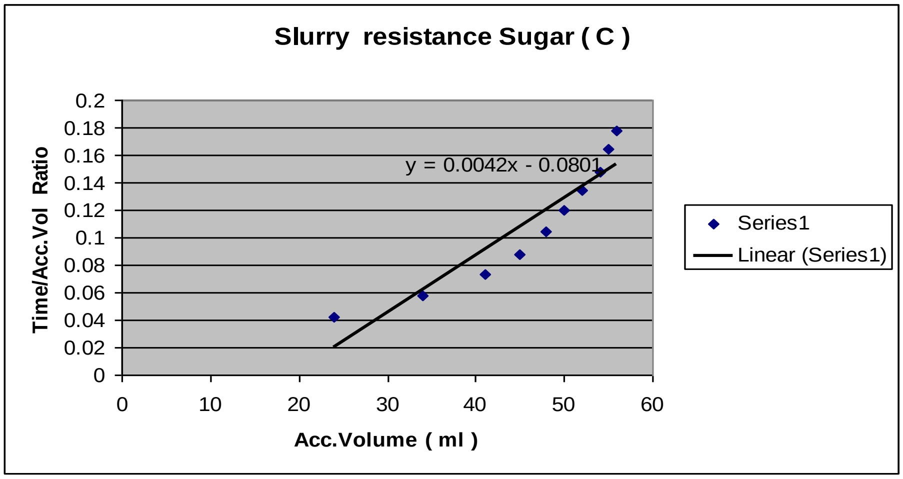

Figure (5): slurry resistance of sugar C

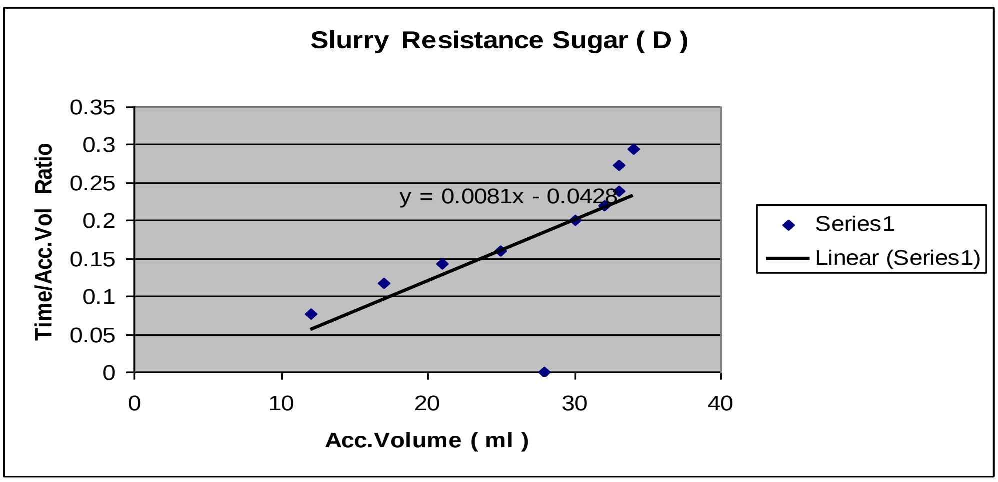

Figure (6) : Slurry resistance of Sugar (D)

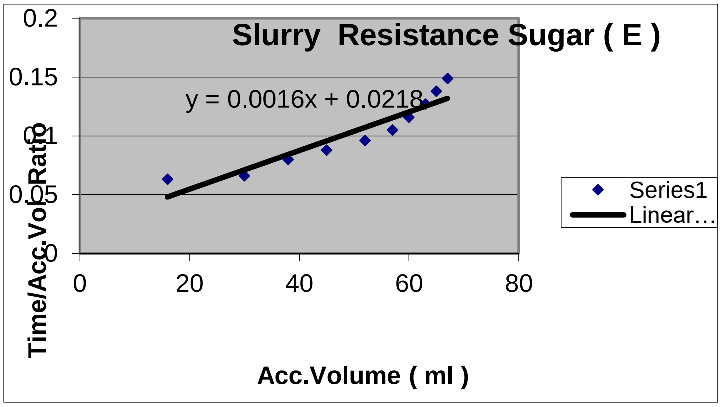

Figure #6 : Slurry resistance of Sugar (E)

Filterability Determination Results of Actual Refinery Carbonated Liquor from VHP &VVHP Sugars :

Samples of carbonated liquor just before the filtration process were collected at 2hrs interval at different occasions ; when the refinery was processing VHP and VVHP sugars .The total collected samples from each sugar were composited every 24–hours , then analysed for the filterability values . The results of the filterability determination for those refinery liquor samples are presented in Table 3. It is clear from this table that the filterability value of the carbonated liquor from VHP sugar was 54 ml in 5 minutes .Whereas, the filterability value for the carbonated liquor from VVHP sugar was 76 ml for the same period of time. The graphical representation for the filterability of actual refinery carbonated liquor from VHP and VVHP sugars is depicted in Figs (8) and (9) respectively . The filterability in both graphs is proportional with the increase in time.

The slurry resistance values for the actual refinery carbonated liquor from VHP and VVHP sugars were presented in Table 4. Those values fall within the optimum range of (1 to 5) as specified by (Field,2001) for a good filtering raw sugar in Australia.

The graphical representation for the slurry resistance of actual refinery carbonated liquors from VHP and VVHP sugars were shown in Figures 10 and 11 respectively .

Table (3): Results of filterability determination for the actual refinery carbonated liquor from VHP and VVHP sugars

Time	Carbonated Liquor (ml)	
(mins)	VHP	VVHP
1	22	32
2	32	47
3	41	59
4	48	68
5	54	76
6	60	80
7	64	87
8	66	93
9	69	98
10	70	102

Table (4) : Slurry Resistance Values of the actual refinery carbonated liquors from VHP and VVHP sugars :

Carbonated Liquor from	Slurry Resistance Value
VHP Sugar	0.74
VVHP Sugar	0.41

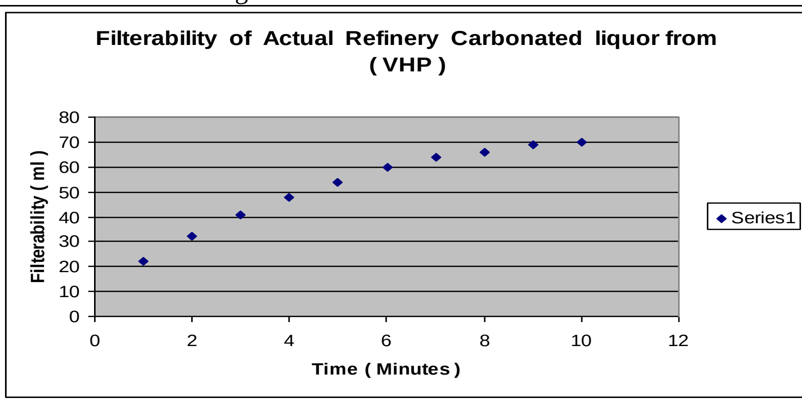

Figure # 8: Filterability for actual refinery carbonated liquor from VHP_sugar

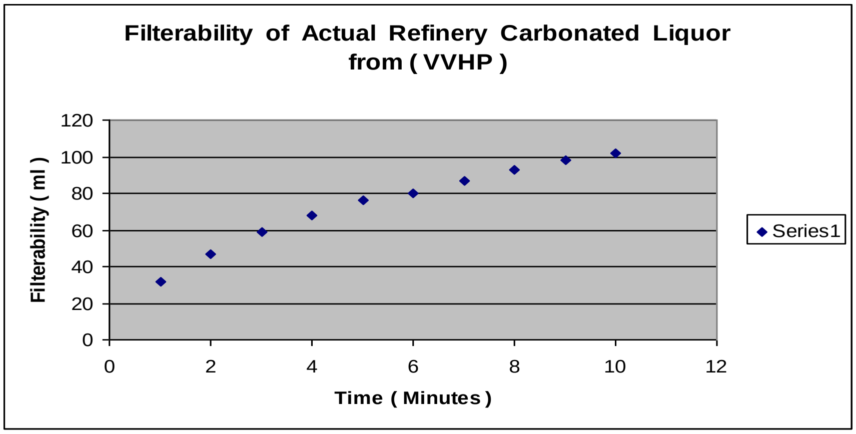

Figure # 9: Filterability for actual refinery carbonated liquor from VVHP sugar

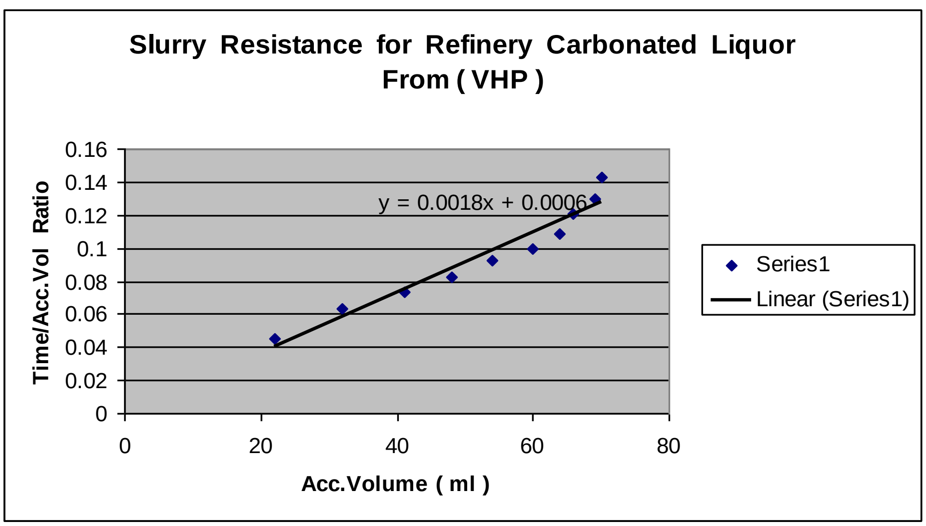

Figure # 10: Slurry resistance for refinery carbonated liquor from VHP raw sugar ,(slope = 0.0018 , SR = 0.74)

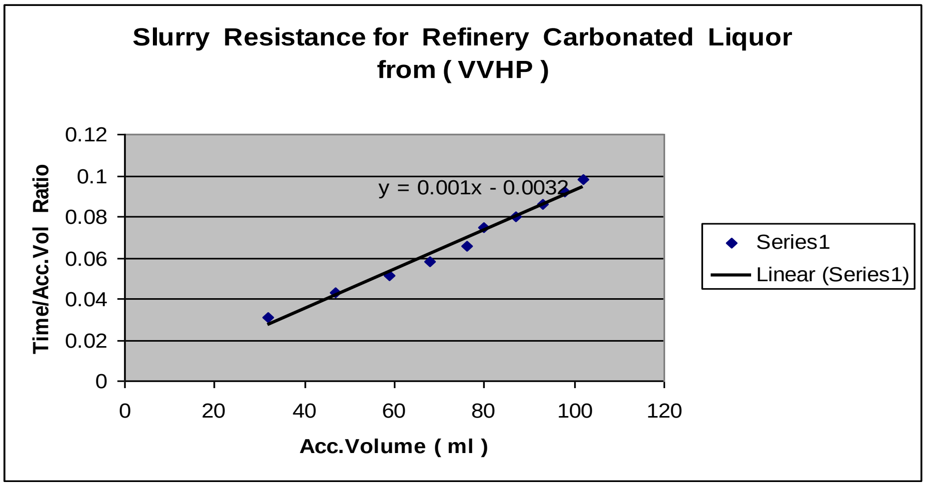

Figure # 11: Slurry resistance for refinery carbonated liquor from VVHP raw sugar (slope = 0.001 , SR = 0.41)

DISCUSSION

Filterability of Raw Sugar Samples in the Laboratory :

Sugar sample –E , which belongs to raw sugar quality group of VVHP gave an excellent filterability , with a value almost similar to the value obtained from white sugar filterability. Such type of sugar is expected to give no problem during the filtration process of the refinery , and all the unit operations will be functioning well with maximum capacity and high efficiency . On the contrary , sugar sample –A ,which belongs to raw sugar quality group of LP , showed a very poor filterability ,with the value of 20 % less than white sugar filterability . Similar type of sugars are expected to experience poor filtration rates , hence reduction in plant capacity and limited outputs . The average filterability value for sugars B ,C , and E as shown in Table 4 , were found to be 55 %

Those sugars belong to raw sugar quality group of VHP. They are expected to show moderate filtration behaviour with good flow rates. Surprisingly , sugar sample

–D which is a VHP raw sugar , gave the filterability value of only 28 % less than white sugar filterability. This value is very close to the filterability of sugar sample–A which is a low pol (LP) .This indicates that raw sugars from different quality groups may filter in the similar manner depending on the effect of quality parameters contributing to the filtration impedance .
Those results were in agreement with the findings of Hidi and Mc Cowage , (1984) who stated that the filterability of low quality raw sugar will approach to 0 % , whereas the filterability of high quality raw sugar will approach to 100 % .

The Prediction of Raw Sugar Filterability Behaviour during the Actual Refinery Filtration Process:

For a better comparison , the laboratory filterability test conditions were kept identical to the actual refinery filtration process conditions . This was very important in order to correlate the laboratory results to the refinery performance . The main difference was that laboratory test was batch process whereas the filtration process in the refinery was continuous .

Figures (8) and (9) indicate the graphical representation for the actual refinery carbonated liquor filtration of VHP and VVHP sugars respectively . The filterability value of the carbonated liquor when processing VHP sugar was 70 ml in 10 minutes , against 102 ml when processing VVHP sugar .This is the typical refinery filtration results after the carbonatation process when the liquor is being filtered using pressure filters coated with the precipitated calcium carbonate obtained from the carbonatation process to act as filter aid .
The average filterability value of carbonated liquor from VHP sugars obtained from laboratory tests was 66 ml in 10 minutes .This value is almost similar to that of actual refinery filtration process (70 ml /10 mins) shown in Table (3).

Likewise, the filterability value for carbonated liquor obtained in the laboratory from VVHP sugar was 98 ml in 10 minutes which is also almost coinciding with the value 102 ml of the actual refinery filtration process. Those results and their graphical representation indicate clear similarity between the laboratory filterability values and the correspondent values of carbonated liquors obtained from the similar qualities of raw sugar in the actual carbonation refinery filtration process . The main reason is possibly attributed to the cumulative effect of raw sugar quality parameters in each quality group and their impact on the filterability. These findings coincide with the previous studies made by Donovan and Lee , (1994) , Simpson and Davis , (1998) , Vianna , (2000) and King ,(2005). All of them agreed that the filterability test done in the laboratory scale can be used to predict the filtration process performance of a carbonation refinery , in particular when the starch threshold level in raw sugar is lower than 200 ppm .

The Prediction of Slurry Resistance Values for Carbonated Liquors during Refining Process:

The average value for slurry resistance of carbonated liquor from VHP sugars in the laboratory was 0.76 ,whereas for the similar quality of raw sugar in the actual refinery filtration process ,as shown in Fig. (10) was 0.74 .These two values are almost the same.

Interestingly , sugar-D which is belonging to the quality group of VHP behaved differently with the slurry resistance value (3.53) .This value is quite high for this group , rather it is closer to the group of low polarization (LP) raw sugars .It has the highest valve of phosphate and its turbidity value is ranked as the second highest .

In the case of carbonated liquor from VVHP sugar , the slurry resistance value obtained in the laboratory was 0.61 , against 0.41 for the actual refinery carbonation. This value is slightly higher than the refinery value .The slurry resistance values of carbonated liquors from VHP and VVHP sugars are quite similar to the actual refinery filtration process .

For the actual refinery carbonation . This value is slightly higher than the refinery value,.7 and .74 Tables 2 and 4 respectivly. The slurry resistance values of carbonated liquors from VHP and VVHP sugars that the laboratory results are quite similar to the actual refinery filtration process .

CONCLUSIONS

The filterability and slurry resistance values for raw sugar samples produced in Thailand , Brazil , South-Africa , Indai , and Sudan were determined using laboratory filteration apparatus.

Interpretation of results obtained from those determinations shows the conclusions below :

1. Raw sugars from the same quality brand may have different filtering behaviour depending on the component influence of their quality parameters.
2. Sugar-A produced in Thailand which is under the raw sugar quality brand of (LP) showed a very poor filterability as in Table 1P12.These results are reasonable for low pol sugar and definitely affect the filtration process.
3. Sugar-B, produced in Brazil gave the highest filterabilty value compared with other sugars from the same quality brand of (VHP) .Whereas , sugar-D produced in India gave the lowest filterability value .
4. Sugar-E , produced in Sudan ,Table2 p14 , gave the lowest value 0.70)of slurry resistance among the VHP sugars .However, it is ranked No.2 as the best filterable sugar.

5. The geographical region did not show clear influence on the filerability of raw sugars produced at that area . Rather, the major parameters concentration within the quality brand was the principal factor determining the filterability.

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دراسة قابلية ترشيح السكر الخام المنتج في مناطق إقليمية وعالمية مختلفة

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الخلاصة

اجريت هذه الدراسة فى مصفى تكرير السكر بدولة الامارات العربية المتحدة بدبى

هذه الدراسة إستهدفت إيجاد العلاقة بين عوامل جودة السكر الخام وقابلية عملية الترشيح. تم إستخدام 5 عينات من السكر الخام تنتمي لمناطق جغرافية متعددة من العالم وتنسب إلي ثلاثة مجموعات جودة مختلفة من السكر وهي ذو نقاوة منخفضة (LP) من تايلاند، ذو نقاوة عالية (VHP) من البرازيل وجنوب أفريقيا والسودان والهند ثم ذو نقاوة عالية جداً (VVHP) من البرازيل تم إستخدام قابلية الترشيح ومقاومة المحلول المرشح كعاملين أساسين للتقييم.

أوضحت نتائج التجارب أن قابلية الترشيح لسكر ذو نقاوة منخفضة تساوي 20% من قابلية ترشيح السكر الأبيض، بينما كانت قابلية الترشيح للسكر ذو النقاوة العالية والعالية جداً تساوي ما بين 80-40% من قابلية ترشيح السكر الأبيض. أما مقاومة المحلول المرشح للسكر ذو النقاوة العالية والعالية جداً في المختبر فكانت (0.76) و(0.64) علي التوالي مقارنة بقيم (0.74)، (0.48) في المصفاة.

كذلك لوحظ أن نتائج قابلية الترشيح ومقاومة المحلول المرشح في المختبر متطابقة مع نتائج قابلية الترشيح ومقاومة المحلول الفعلية في المصفاة مما يدل علي أنه يمكن إستخدامها كوسيلة للتكهن بخصائص الترشيح لأنواع السكر الخام من نفس مجموعة الجودة.