MODIFIED SINGLE SULFITATION PROCESS FOR PRODUCING BETTER QUALITY PLANTATION WHITE SUGAR

By

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Abstract

THE PRESENCE of sulfur in sugar, apart from making it potentially harmful for human consumption, causes the sugar to deteriorate at a faster rate during storage. These issues and the steep rise in sulfur prices, albeit temporary, have brought to the fore the need to reduce the sulfur dosage used in the production of plantation white sugar (PWS). The process generally used in the production of PWS (in addition to juice sulfitation) is syrup sulfitation, which reduces sugar colour by a further 6–15%. However, this reduction in sugar colour is short-lived and it also reduces the pH of the syrup, with the result that subsequent manufacturing processes are carried out under acidic conditions. Because of this, there could be significant sugar losses from sucrose inversion. The use of suitable biocides for cane and mill sanitation has been shown to reduce the acidity of the mixed juice, as also the quantum of the high molecular weight polymeric compounds in the sugar and the molasses. In view of these observations, a new process (the VMK process) was developed to produce PWS without resorting to syrup sulfitation, namely, a single sulfitation process using a polymer together with appropriate biocides that reduce impurities and colour by about 12–15%. This process was tried in a full season of about 100 days in a 2500 tcd factory where 25 000 t of sugar was produced with colour below 80 IU. Further improvement in sugar quality was also achieved with the help of an on-line colour monitoring unit, WG Colormet 1.0, as well as through tight control on chemical consumption, especially that of lime and sulfur. The quality of the sugar, which was consistently achieved, showed colour to be <55 IU, conductivity ash <0.015%, calcium <30 ppm, pol >99.85, and dextran and sulfur below detectable limits. The colour transfer index was found to be 130:1 from clear juice to final sugar, as against the reported index of 100:1 from A massecuit to sugar. Based on these findings, a patent has been filed for the VMK process for the production of good quality PWS.

Introduction

Sulfur dioxide (SO₂) is a toxic gas. When it is used during the manufacture of any food product, residual traces of sulfur remaining in such food product may be hazardous to human health. Hence the use of SO₂ is always restricted. The food industry does not permit or recommend the use of any sulfur compounds for food preservation.

In the sulfitation process, the use of the sulfur burner is difficult as it causes the toxic SO₂ to be released into the atmosphere, leading to serious environmental pollution because of this potent greenhouse gas. It can also create health problems for the workers handling the sulfur burners.

Although the sugar manufactured through syrup sulfitation has low colour and good ‘lustre’ initially, it loses both the colour and the ‘lustre’ within a few months. Depending on storage conditions, the colour can increase from 60 IU to 100 IU or even 120 IU within 6 to 10 months. There are several reasons for this deterioration, but one of the main reasons is sulfur. When first
applied, SO₂ bleaches the syrup, reduces the iron present in the juice and makes it colourless, resulting in a low colour sugar. However, this is only a temporary effect. The colour returns with time, as the remaining iron compounds oxidise into dark-coloured compounds. In addition, the sugar retains high sulfur content.

Sulfur dioxide is first used to treat the juice, which helps in the clarification process. Then it is again used in syrup treatment. The latter treatment is known to reduce the viscosity of the syrup / massecuite and improve molasses exhaustion. It also reduces the pH to pH 5.5 or less depending on how much SO₂ is used. Hence, the crystallisation process is carried out in an acidic environment; this favours sucrose inversion, which, in turn, affects sugar recovery. The quantum of sulfur in the sugar produced in good sugar factories is about 15 ppm (the highest permissible level), but it can be over 25 ppm in the sugar produced by others.

The treatment of the syrup with SO₂ is known also to reduce sugar colour by just 6–15% (Keskar, 2009, pers. comm.; Londhe, 2009, pers. comm.). However, significant amounts of sulfur end up in the molasses which may be used for producing alcohol by yeast fermentation. Sulfur affects the fermentation of the molasses by inhibiting the growth of the yeast. Moreover, traces of the sulfur that get into the alcohol make it unsuitable for use as a fuel supplement, as also for use in the liquor and pharmaceutical industries, unless further refinements are carried out to remove these impurities.

Thus the use of SO₂ in syrup does not provide any significant benefit. Further, whatever benefit does result lasts only for a short period of time. This realisation prompted the development of an alternative technology that would avoid at least syrup sulfitation and help in producing good quality sugar.

The use of appropriate biocides in proper doses is known to limit the microbial degradation of mixed juice and hence limit any reduction in the acidity of the primary and mixed juice and prevent the formation of degradation products like polysaccharides (Kulkarni, 2004). Further, the need for additional lime is prevented and the level of residual calcium as soluble organic calcium salts in the clear juice is not increased. Similarly, there is some reduction in the rise in the colour of juices (Kulkarni and Warne, 2004).

Any reduction in polysaccharide content can play a significant role in molasses exhaustion, especially when syrup sulfitation is avoided. The use of appropriate biocides in the process can control the rise in this polysaccharide content; however, polysaccharides already present in the sugarcane and formed during the cut-to-crush delay will obviously get concentrated. The use of a mixture of crystal mobilisers, crystal developers and viscosity reducers can help to improve the pan output, as well as in better exhaustion. Such a formulation is also known to reduce sugar colour by about 12% (Kulkarni and Pallod, 2005). Thus, even when syrup sulfitation is avoided, its benefits can be more than matched by using appropriate biocides.

**New single sulfitation process—VMK process**

A new process has been developed, which requires only a slight modification to the existing process. It seeks to prevent the entry of impurities into the process. This is done by using appropriate chemicals in proper doses at the right stages of the process. Some of the currently used chemicals are replaced in the new process with more appropriate chemicals while others are new as identified in Table 1. Apart from the changes in the chemicals, the only modification needed in the process is the isolation and shutting down of the sulfur burner for the syrup.

**Chemicals used in the VMK Process**

Polmax Supreme is used for cane sanitation by spraying onto prepared cane at 2–5 ppm before the cane enters the fiberisor hammer. It consists of various salts of methyl and ethyl dithiocarbamates, organo-sulfur based activator and penetrating agents and has an active ingredient concentration of 40%. Up to 90% of the microbes are killed in just one minute of the spraying if Polmax Supreme is sprayed onto the cane at 10 ppm.
Polmax ESR is used for mill sanitation. It is added continuously at 10 ppm with the application split between all mills after the 2nd mill. It is a chemical formulation of various salts of methyl and ethyl dithio-carbamates and has an active ingredient concentration of 40%. At a dosage of 10 ppm, 90% of the microbes in the cane juice are killed in 10 minutes.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Normal process</th>
<th>VMK process</th>
<th>Cost implication</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cane sanitation</td>
<td>Not used</td>
<td>Polmax Supreme @ 5 ppm</td>
<td>+USD30 000*</td>
</tr>
<tr>
<td>Mill sanitation</td>
<td>About USD20 000 Carbamate @ 10 ppm</td>
<td>Polmax ESR @ 10 ppm, USD29 000</td>
<td>+USD9000*</td>
</tr>
<tr>
<td>Lime</td>
<td>Used based on experience</td>
<td>Dose specified as per results</td>
<td>Could be reduced</td>
</tr>
<tr>
<td>Phosphoric acid</td>
<td>Used arbitrarily</td>
<td>Used judiciously (Not used)</td>
<td>Can be reduced</td>
</tr>
<tr>
<td>Flocculating agent</td>
<td>Magnafloc LT27 @ 1–2 ppm</td>
<td>No change</td>
<td>No change</td>
</tr>
<tr>
<td>Sulfur</td>
<td>0.07% or more (700 t)</td>
<td>0.03% saving about 300 t</td>
<td>– USD45 000**</td>
</tr>
<tr>
<td>Special agents</td>
<td>Colour precipitant, USD7 /kg. @ 10 ppm</td>
<td>Sucolor @ 10 ppm, US$ 7 /kg</td>
<td>+ USD70 000* May not change</td>
</tr>
<tr>
<td>Viscosity reducer</td>
<td>Unspecified surfactant 2–5 ppm</td>
<td>New formulation 2–5 ppm</td>
<td>+ USD20 000</td>
</tr>
<tr>
<td>Colour coagulant</td>
<td>Used to improve colour</td>
<td>Not used</td>
<td>?</td>
</tr>
</tbody>
</table>

* Prices are calculated at maximum use of chemicals for the VMK Process, actual use of chemicals could be less depending on factory conditions.

** Saving is more as sulfur consumption is less than 0.017%

Sucolor is used as an impurity-removing agent. It is added continuously at 10 ppm in the clear juice receiving tank. It is a special polymer of coco-di methyl amine, which reduces the affinity of various impurities to attach themselves to the sucrose crystal lattice during growth. Thus the colour transfer index is improved to produce lower coloured sugar.

Viscosity reducer is used to improve crystallisation and improve crystal washing efficiency. It is a formulation of various glycols, oleates, acetates with surfactants. It is added continuously at 2–5 ppm to the syrup.

Thus there would be some rise in the cost of process chemicals used for the VMK process as compared to the normal double sulfitation process for factories that do not use special agents like colour coagulants and/or colour precipitants. However, considering the benefits of lower inversion losses of sucrose, the VMK process offers economical advantages over the normal process.

**Experiment 1**

Initially, during the season 2006–2007, trials were successfully carried out for 15 days, which were repeated for 45 days, in a 3500 tcd factory, the Kisan Veer Statra S.S.K. Ltd., at Bhuinj, near Satara.

There was noticeable improvement in the process with respect to steam consumption, quality of bold grain sugar and bagasse savings. The viscosity reduction assisted to reduce the boiling time of A strikes by 10 minutes and B strikes by 20–30 minutes. About 135 000 bags of sugar were produced without syrup sulfitation.

This sugar was sent for analysis to Maarc Labs, Pune (an ISO 17025 lab accredited by NABL). Their report clearly states that the quality of the sugar produced by this process was very good. The SO2 content and dextran content were below detectable levels and it had a microbial count of less than 120 cfu per 10 g. The conductivity was only 0.014%, and the moisture and calcium levels were lower. This substantial reduction in most of the impurities as compared to those found in the normal PWS has also improved the sucrose content to 99.85% pol.
Sugar of such quality has been produced for the first time in India without the use of any additional equipment or any modification to the process for clarifying the juice and syrup. It is also noticed that the pH of the sugar was more than 6.6. Table 2 gives the requirements for PWS as per IS specifications, EU II specifications and the results obtained by using the VMK process.

**Table 2—Specifications of sugar and comparison with the VMK process.**

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>IS 5982–2003</th>
<th>Codex 2004</th>
<th>VMK process</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum moisture, %</td>
<td>0.10</td>
<td>0.06</td>
<td>0.0156</td>
</tr>
<tr>
<td>Minimum sucrose, %</td>
<td>99.5</td>
<td>99.7</td>
<td>99.87</td>
</tr>
<tr>
<td>Maximum reducing sugars, %</td>
<td>0.10</td>
<td>0.04</td>
<td>0.01</td>
</tr>
<tr>
<td>Maximum colour, IU</td>
<td>150</td>
<td>45</td>
<td>70</td>
</tr>
<tr>
<td>Maximum conductivity ash, %</td>
<td>0.1</td>
<td>0.027</td>
<td>0.0146</td>
</tr>
<tr>
<td>Maximum sulfur dioxide, mg/kg</td>
<td>70</td>
<td>15</td>
<td>&gt;5</td>
</tr>
<tr>
<td>Maximum lead, ppm</td>
<td>5.0</td>
<td>1.0</td>
<td>ND</td>
</tr>
</tbody>
</table>

**Experiment 2**

The same factory, Kisanveer Satara SSK Ltd., produced raw sugar during the season 2007-2008 and similar trials were conducted for a week to produce raw sugar without the use of sulfur. The sugar looked good and also had ‘lustre’, with a colour of 170 IU. Although the colour was higher than the IS specifications for PWS, the raw sugar looked better than one year old PWS from some factories. The raw sugar was sent for analysis at Savola Sugars, KSA, which revealed that it was better than the best raw sugar developed in Brazil (Table 3), especially with respect to conductivity ash and some other impurities. This sugar is now called pre-refined (PR) sugar. It is best suited as raw material for sugar refineries for producing refined sugar, with minimal difficulties and at low cost.

**Table 3—Specifications of various international raw sugars and a new specification for pre-refined sugar from VMK process.**

<table>
<thead>
<tr>
<th>VHP</th>
<th>VVHP</th>
<th>QHP</th>
<th>EHQ</th>
<th>PR sugar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pol</td>
<td>99.2</td>
<td>99.6</td>
<td>99.6</td>
<td>99.7</td>
</tr>
<tr>
<td>Colour, IU</td>
<td>1500</td>
<td>450</td>
<td>700</td>
<td>350</td>
</tr>
<tr>
<td>Ash, %</td>
<td>0.20</td>
<td>0.15</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>Invert, %</td>
<td>0.25</td>
<td>0.15</td>
<td>0.15</td>
<td>0.1</td>
</tr>
<tr>
<td>Starch, mg/L</td>
<td>250</td>
<td>110</td>
<td>50</td>
<td>100</td>
</tr>
<tr>
<td>Insoluble solids, mg/L</td>
<td>250</td>
<td>150</td>
<td>100</td>
<td>50</td>
</tr>
</tbody>
</table>

**Experiment 3**

The VMK process with single sulfitation was also tested at a 2500 tcd private factory, Cane Agro Energy Ltd., Dongari near Karad for the entire 2007–2008 season. A WG Colormet 1.0 instrument was used to display online the colour level of the clear juice every 10 seconds. This helped in the optimisation of chemicals used for the clarification.

The optimisation of the process resulted in production of sugar with colour less than 60 IU for about a month. Sometimes, a colour of 50 IU was also recorded. During this optimisation of the clarifier it was observed that, depending upon cane quality; the lime quantity could be reduced and therefore sulfur quantity in achieving better colour removal. Hot liming was used with simultaneous sulfitation, whereby the quantity of lime was reduced, with a concomitant reduction in sulfur, to maintain pH 6.9 in the clear juice. This adjustment was based on the observation of clear juice colour on the WG Colormet 1.0.
These observations were then used to reduce the lime quantity and thus sulfur quantity, in order to maintain the colour of the clear juice at around 8000 IU. The lime and sulfur quantities used were the lowest for the final three weeks of the season (days 65 to 80 in Figures 1 and 2).

The colour of the clear juice was around 7500–8500 IU and the sugar colour was often below 60 IU, especially in the final three weeks.

Figure 1 shows that the colour transfer index increased from a typical 100:1 to more than 130:1 during the last three weeks of the season. This could be due to the better clarification, as well as the better process control assisted by the online monitoring of the clear juice colour. This resulted in the reduction in sulfur consumption to less than 0.017% on cane, yet sugar with less than 60 IU colour was produced.

Figure 2 shows the daily average sugar colour for the season. Figure 3 shows the daily average sugar colour (×100) and clear juice colour during experiment 3.
Conclusion

Good quality sugar can be produced by avoiding syrup sulfitation without entailing any additional capital expenditure on equipment etc or any alterations or modifications in the process. Sulfur consumption can be reduced still further by using the WG Colormet 1.0 to monitor online the colour of the clear juice. Depending on the cane quality, sulfur consumption on cane can be reduced to less than 0.02%. In short, the VMK process can be adopted for producing both PWS and raw sugar. For PWS, the VMK process uses single sulfitation while, for raw sugar, it dispenses with the use of sulfur altogether.

The quality of the PWS as well as the raw sugar produced by the VMK Process is superior to that of the normal PWS and raw sugar respectively. The lower sulfur consumption and, consequently, the lower residual sulfur content in the sugar means that the keeping quality of the sugar is superior to the PWS sugar.

Acknowledgements

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SULFITAGE SIMPLE MODIFIÉ POUR LA PRODUCTION DE SUCRE BLANC PLANTATION AVEC UNE MEILLEURE QUALITÉ

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MOTS-CLES: Sulfitage, Soufre, Sucre Blanc de Plantation, Impuretés, Processus VMK.

Résumé

LA PRESENCE du soufre dans le sucre le rend potentiellement nuisible à la consommation humaine et entraîne le sucre à se détériorer plus rapidement au cours du stockage. Ces questions et la forte hausse des prix du soufre, quoique temporaire, demandent la réduction de la dose de soufre utilisée pour la production de sucre blanc plantation (PWS). Le processus généralement utilisé dans la production du PWS (en plus du sulfitage du jus) est le sulfitage du sirop, ce qui augmente la décoloration du sucre par 6–15%. Toutefois, cette réduction en couleur de sucre est de courte durée; elle réduit également le pH du sirop, de sorte que les procédés de fabrications ultérieures sont effectués en milieu acide. De ce fait, il pourrait y avoir des pertes de sucre significatives grâce à l'inversion du saccharose. L'utilisation de biocides appropriés pour l'assainissement des cannes et des moulin réduit l'acidité du jus, comme aussi le quantum des composés polymères de haut poids moléculaire dans le sucre et la mélasse. Compte tenu de ces observations, un nouveau processus (le processus VMK) a été développé afin de produire le PWS sans avoir recours au sulfitage du sirop, à savoir, un processus de sulfitage simple à l'aide d'un polymère avec biocides appropriés qui réduisent les impuretés et les couleurs d'environ 12 à 15%. Ce processus a été utilise pendant une saison complète d'environ 100 jours dans une usine broyant 2500 tonnes de canne par jour; on a produit 25 000 tonnes de sucre avec une couleur inférieure à 80 UI. On a également obtenu une amélioration de la qualité du sucre à l'aide d'un appareil WG Colormet 1.0 surveillant la couleur en ligne, ainsi que par le contrôle strict sur la consommation de produits chimiques, en particulier celui de la chaux et du soufre. La qualité du sucre, qui a été constamment obtenue, a montré une couleur à 55 UI, une conductivité (basée sur les cendres) de 0.015 %, un calcium de 30 ppm, un pol > 99.85; le dextran et le soufre n’ont pas été détectés. L'indice de transfert de couleur a été 130:1 à partir du jus clair au sucre final, par rapport à l'index signalé de 100:1 d'une massicuite au sucre. Selon ces conclusions, un brevet a été déposé pour le processus de VMK pour la production de PWS d’une bonne qualité.
PROCESO MODIFICADO DE SULFITACIÓN SIMPLE PARA PRODUCIR AZÚCAR BLANCO DE MEJOR CALIDAD

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Resumen

La presencia de azufre en el azúcar, además de hacerlo potencialmente nocivo para consumo humano, causa que el azúcar se deteriore a una tasa más alta durante el almacenamiento. Estos aspectos junto con el incremento en los precios del azufre, han resaltado la necesidad de reducir las dosis de azufre usadas en la producción de azúcar blanco. El proceso generalmente seguido, además de la sulfitación de jugo, es la sulfitación de meladura, la cual reduce el color un 6–15% adicional. Sin embargo, esta reducción es de corta vida y también reduce el pH de la meladura con el inconveniente que las etapas posteriores de proceso se realizan bajo condiciones ácidas. Debido a esto pueden existir pérdidas importantes por inversión de sacarosa. El uso de biocidas adecuados y sanitización de molinos han mostrado reducciones en la acidez del jugo diluido así como de la cantidad de compuestos poliméricos de alto peso molecular en azúcar y mieles. Un nuevo proceso (proceso VMK) fue desarrollado para producir azúcar blanco sin recurrir a la sulfitación de meladura, es decir, un proceso de una sola etapa, usando un polímero junto con biocidas apropiados que reducen impurezas y color en cerca de 12–15%. Este proceso fue ensayado en una zafra de cerca de 100 días en una fábrica de 2500 tcd, donde se produjeron 25 000 t de azúcar con color por debajo de 80 IU. Un mejoramiento adicional de la calidad del azúcar se obtuvo con la ayuda de una unidad de monitoreo continuo de color, WG Colormer 1.0, así como con cuidadoso control del consumo de químicos, especialmente de cal y azufre. La calidad del azúcar, lograda en forma sostenida, <55 IU, conductividad de cenizas <0.015%, calcio <30 ppm, pol >99.85, y dextranas y azufre por debajo de los límites detectables. El índice de transferencia de color se encontró como 130:1 desde jugo claro hasta azúcar final, frente al índice reportado de 100:1 desde masa A hasta azúcar. Con base en estos hallazgos se ha solicitado patente para el proceso VMK para la producción de azúcar blanco de buena calidad.