

DEVELOPMENT OF AN ETHANOL PRODUCTION PROCESS WITH STILLAGE RECYCLING AT PILOT PLANT-SCALE.

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Abstract

The production of energy from biomass has been object of wide research in the last years, especially the production of fuel alcohol, in Colombia, has increased thanks to laws promulgated by the government; parallel to this the generation of residues increases also, in this case called stillage or vinasse, for a conventional process 13 to 15 liters are obtained of stillage by liter of ethanol, which for the quantity of ethanol projected, of 750 million liters/year of alcohol in this country, would be generated between 9750 and 11250 million liters of stillage/year, a so big quantity. An ethanol production process with stillage recycling in bench-scale was developed in order to determine stillage percentage recirculation and the number of successive recycles that can be carried out, without detriment in the ethanol productivity achieved. With the best results obtained in bench-scale will be climbed to pilot plant-scale. The inhibitors presents in the stillage, as acetic acid and solids, which increase by accumulation, with the continuous recycles, limit the recirculate percentage and the number of recycles that can be carried out, for this reason several alternatives were proposed and evaluated in order to remove them or diminish their harmful effect.

Keywords: stillage recycling, ethanol production, clean technology, pilot plant scale.

1. Introduction

The great demand of alcohol that actually we can observe causes, as response of the industry, a high production with the subsequent generation of residues as the vinasse or stillage, to which it is necessary to give her the importance that is deserved, as pollutant potential or source of by-products if this way it is wished.

As answer to the world critical environmental situation, to the gradual decrease of the fossil fuels and the possibility in the country of developing alternative energies using renewable resources, the government determined to send the Law 693 of 2001, where is regulated the addition of anhydrous ethanol to the gasolines in 10% starting from 2006, according to this it is necessary to increase the production of this alcohol, until levels initials of 750 million liters / year, (Program of Carburating Alcohols).

As agricultural sources for the obtaining of the ethanol there are the sugary ones (cane of sugar, beet), the starch sources (barley, banana tree, yucca), the lignocellulosics (wood, cotton). The physicochemical characteristic of all sources are different whose causes differences in the adaptation operations for a common later treatment in the three cases, whichever it is the source of sugar for the fermentation, the processes have the high production of liquid effluents of the distillation in common, denominated in this particular case, vinasses or stillage, Durán de Bazúa in 1993 defined the vinasse like "a dilution of substances like mineral and organic salts with relative value and with potential for diverse uses", thanks to his organic high load (6.5.7.6%), CaO (4.57%) and K₂O (1.06-1.53%) (GEPLACEA / PNUD / ICIDCA 1990), among other substances, that make it appropriate of being used as energy source, protein source, mineral salts source for fertilizers and concentrate for animals.

In the conventional process of production of alcohol the vinasse production oscillates between 13 and 15 l/l ethanol, with this and if there are a requirement of 2 million liters of ethanol / day approximately, according to the Carburating Program of Alcohol, the quantity of the effluent per day could ascend to 30 million liters, that it is an important quantity and that could harm a lot in the environment, if it isn't managed appropriately like some evidences show us for example the experiences of the Cauca's river Valley, that which can provoke the cancellation of the Environmental License in these distilleries.

As alternative to reduce the vinasse volume has been proposed the recirculation (recycling) from the same ones to the fermentation stage, like dilution water, however the presence of inhibitors like the acetic acid, limits their application. The present work evaluates the effect of the recirculation on the alcoholic yield and biomass generation in the fermentation.

2. Materials and Methods

2.1 Culture

The used culture was a *Saccharomyces cerevisiae* ATCC26603, donated by the Institute of Biotechnology of the National University of Colombia (IBUN), in lyophilized state, it was resuspended in peptoned water

and it was sowed in a growth medium (yeast extract 1%, peptone 2%, glucose 1%, maltose 1%, agar 2%, water 93%, all percentages w/w) at 30°C for 48 h, starting from this cultivation broth was prepared the main cultivation (peptone 1%, sucrose 2%, K₂HPO₄ 0.2%, MgSO₄ 0.001%, (NH₄)₂SO₄ 0.2%, water 96.5%, pH 4.5) and was inoculated with 15% of the previous broth and it was renewed monthly.

2.2 Fermentation and distillation

The inoculum (4.5 L) for the fermentation, constituted 15% of the broth of fermentation (30 L), both were compound of cane molasses 16.5%, water 83.5%, (NH₄)₂SO₄ 0.01%, the pH was adjusted at 4.5, with a solution 15% w/w of H₂SO₄, the time of preparation of the inoculum was of 14 h and that of the fermentation of 28 h in a CE 640 Fermentation & Alcohol Production Pilot Plant / PC (Gunt, Hamburg) with temperature control at 30°C and 110 rpm, after the fermentation time, the fermented must was pumped to the distillation unit, there was carry out the thermal process during 6 hours with a vapor pressure of 250 kPa.

2.3 Vinasse treatment

The vinasse remains in the boiler after the distillation, then it's carry to a continuous centrifuge at 1200 g, the light phase in then ready to be recycled, replacing 70% of the dilution wáter.

2.7 Analysis

The kinetics of the fermentation was verified by analysis of sugars and etanol at different fermentation times by high performance liquid chromatography (HPLC) in a Waters chromatography system, with a detector of refraction index, each sample was centrifuged 900 g during 15 minutes, later it was diluted ten times, starting from the second fermentation a solid phase extraction was applied using Sep pack C18, to retire the extractant agent's residuals that could remain in the recycled vinasse, after the sample was filtered using a 0.22 μm membrane and finally it was injected manually in the cromatograph. The used column was a Sugar-Pack type Waters, as mobile phase it was used water deionized and degasified with a flow of 0.5 ml / min, the column temperatures and detector were respectively of 84 and 40°C, the volume of sample was 20 μl .

The biomass was determined, using the technique of the camera of Neubauer.

3. Results and Analysis

The fermentative capacity of the yeast was evaluated previously (Castro, 2006) and in this work was observed the soluble solids decrease with the fermentation time in the CE 640 Fermentation & Alcohol Production Pilot Plant / PC (Gunt, Hamburg), the results are observed in the figure 1.

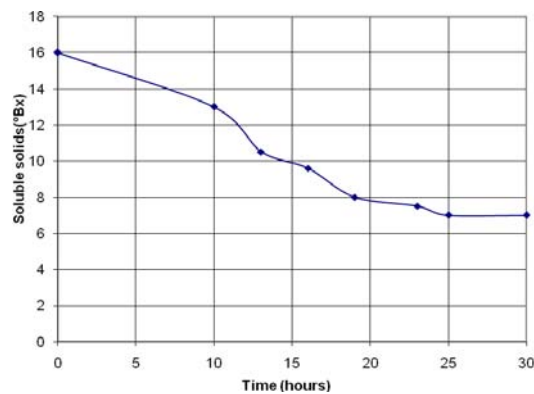


Figure 1 Soluble solid decrease with the fermentation time

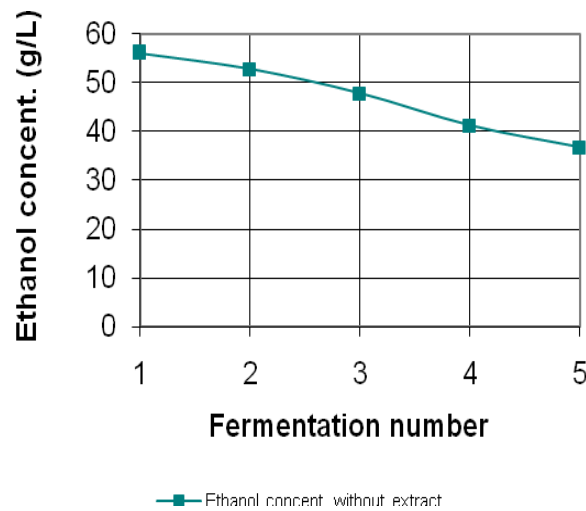


Figure 2 Ethanol final concentration of the fermented musts prepared with recycled vinasse

The figure 2 shows how varies the concentration of ethanol in each one of the five fermentations in series, with recirculation of 70% of the vinasse only applying the centrifugation, is possible to see a similar behavior in the work of Castro et. al. at 2006 but with a smaller final ethanol concentration, of 36.8 g/L front 40.2% of the previous work.

The figure 3 shows something similar to the figure 2 to the ethanol, this time with the biomass, in this case it presents a continuous descent, making notorious that the answer of the yeast is more sensitive than that of production of ethanol, in the first case the reduction was of 80%, in thesecond only of 40%. The results are very close to that obtained by Castro(2006),

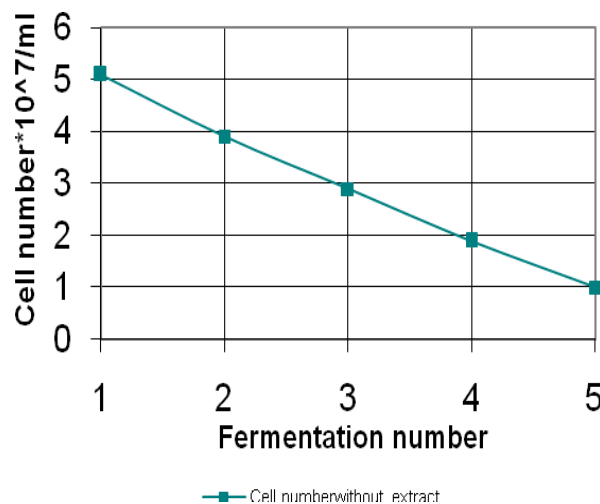


Figure 3 Final biomass in the fermented musts prepared with recycled vinasse.

Besides the inhibitory effect for the fermentation by-products, is very important the effect of the increase of the solids in the vinasse, they can include the not fermentable sugars that remain in the vinasse after the fermentation and the distillation, mineral salts not assimilated by the yeast, are also included, these substances are increased with the

advance of the serial fermentations, due to the molasses and the nutrients with those the mediums of fermentation are prepared are rich in these substances (Maiorella, 1984), others are by-products of the metabolism of the yeast like glicerol, propanol, furfural and lactic acid that also inhibit the fermentation and the speed of growth (Navarro, 1989 and Maiorella 1983).

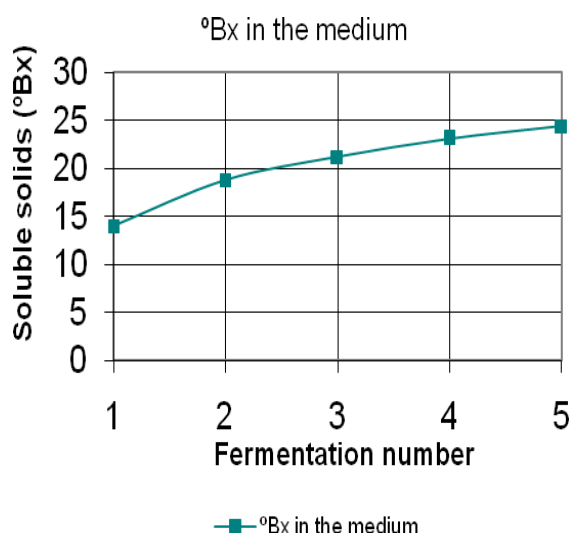


Figure 4 Increase in the soluble solids in the médiums prepared with recycled vinasse

In the figure 4 the increase of solids is observed in the mediums that were prepared with the recycled vinasse, the soluble solids (fermentable coming from the molasses and not fermentable coming from the vinasses) in the mediums of the fifth fermentation, reached values of 25°Bx, very close at the toxicity level, of 26% of dry matter presented for Navarro (2000), knowing that of the total of dry matter in the vinasse the 85 to 90% constitute the soluble solids, being this another of the possible reasons for the decrease in the productivity of ethanol and generation of biomass in the fifth fermentation and still for the fourth, where the values of dry matter are from the order of 23°Bx that are very near to the toxicity limit value, provoking the same effect on the two mentioned answer variables. Thatipamala and collaborators in 1992, reached a similar conclusion, since they verified a decrease of 33% (w/w) in the production of ethanol, when the content of solids in the fermentation medium increases from 14% to 27% (w/w).

4. Conclusions

The results obtained in laboratory scale are very similar to that in the pilot plant scale.

The fermentation time can be from 20 to 30 hours depending on the scale used.

The ethanol concentration decrease 40% and the biomass 80% with the the five fermentation in series, the principal reason could be the increase in the soluble solids, in this case of 40% aproximately.

During the five fermentations it was achieved a saving of water of 48% and a decrease of the volume of vinasses of 40%.

5. References

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