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# RESEARCHES REGARDING INORGANIC ACCELERATORS USING IN POMACE FERMENTING PROCESS

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## Abstract

*The grape pomace represents one of by-products from wine technological process with multiples valorization possibilities. One of these directions is represented by ethylic alcohol obtaining. This paper presents researches regarding inorganic accelerators using in grape pomace fermenting process. Anaerobic digestion process inside carbohydrates biodegradation from grape pomace may be augmented with $(NH_4)_3PO_4$ as process accelerator. The obtaining results lead to the following conclusions: supplying with 2.0-2.5 % $(NH_4)_3PO_4$, 98% concentration, has lead reducing of biodegradation time with 32%; in the end of the process, the residual composition of the grape pomace is the following: 1.07-1.44% N, 1.0-1.83% $P_2O_5$, 0.65-0.88% $K_2O$, 6.02-8.9% $CaCO_3$, pH of aqueous extract is 6-6.9 and C/N proportion is 17.8-18.2.*
***Keywords**: grape pomace, inorganic accelerators, anaerobic digestion process, valorization, wastes, ethylic alcohol*

## Introduction

Grape growing and wine making generates a number of wastes and by-product. These materials include vine pruning, grape stalks, grape pomace and grape seeds, yeast lees, tartrate, carbon dioxide and wastewater (Hazell, 2000). Grape pomace can be used for alcoholic beverage production.

In past farmers had a number of agricultural activities permitting them to "recycle" wastes and byproducts from grape and wine production. Tradition, experience and "common rural sense" had taught farmers that there is nothing to waste. Every product would become fertilizer, animal feed or fuel (Bertran, 2004, Famuyiwa, 1981).

Today's grape growers and winemakers specialize in just one sector. Industrialized production demand higher production volumes and the use of traditional byproducts were replaced by commercial products of low cost and higher efficiency. The new production methods along with the increase in number of wineries and vineyards as well as the increase in production volume, results in an exponential increase of wastes. The "rural wisdom" was forgotten.

As legal, environmental and economic issues are being reconsidered in the past two decades (Hazell, 2000; Bisson, 2002), it becomes more and more obvious that disposal and landfill of those wastes present environmental and social drawbacks. New technologies were proposed not only for their re-use in agriculture, but also for the production of common and novel products for other sectors. For example, using solid state grape pomace fermentation we can produce a variety of compounds like ethanol, citric acid, gluconic acid etc. (Brega, 2002, Hong, 1988, Leber, 2004, Louli, 2004).

The interest on developing product and process for winery residues is increasing and this is evident from the number of scientific publications and researches (Eioion, 1999).

One of this researches is the one presented below, who aims to establish the dominant factors which influence the anaerobic digestion of the pomace using inorganic process accelerators such as $(NH_4)_3PO_4$. It must be said that, in order to use any technique on an industrial scale, it is necessary to undertake laboratory and pilot trials, life cycle analysis and feasibility studies.

## Experimental

Grape pomace used in the determinations has a humidity of about 60% and makes up for 11-15% of grapes crushed during the wine making process. One tone of pomace is composed of 249 kg of stalks, 225 kg of grape seeds and 425 kg of grape pellicles. The medium grape pomace chemical composition is presented in table 1.

The anaerobic conditions for the fermenting process were assured by the design of the storage and fermentation installation. A polyethylene material, 2 mm thick, was used for the lateral and superior protection of the installation, for the inferior part the insulation being made by the earth support of the installation.

**Table 1.** Grape pomace chemical composition

| Component | Values, % |
|---|---|
| Water content | 57.7 |
| Alcohol content | 3.34 |
| Ash content | 2.55 |
| Nitrogen content | 0.924 |
| Alcohol soluble substances content | 4.51 |
| Cellulose content | 31.58 |
| Maximum sugar content | 10 |

In order to increase the fermentation process yields, thus reducing the biodegradation time, we used as a process accelerators $(NH_4)_3PO_4$. The experimental program was conceived with the utilization of programming model of experiences in the centered system by second degree, having four independent variables and 31 experiments. The independent parameters which conduct and define the accelerated anaerobic digestion of the grape pomace are presented in table 2.

**Table 2.** Experimental conditions

| Independent variables | Independent parameters values | | | | | | |
|---|---|---|---|---|---|---|---|
| | X – codified values | | | | | | |
| | $X_i$ | -2 | -1 | 0 | 1 | 2 | Δ x |
| | Actual values | | | | | | |
| Grape pomace toatl sugar content, % | $X_1$ | 1 | 2 | 3 | 4 | 5 | 1 |
| $(NH_4)_3PO_4$, % | $X_2$ | 0.5 | 0.75 | 1.0 | 1.25 | 1.5 | 0.25 |
| Digestion time, days | $X_3$ | 24 | 48 | 72 | 96 | 120 | 24 |
| Grape pomace humidity, % | $X_4$ | 45 | 50 | 55 | 60 | 65 | 5 |

## Results and Discussions

The dependent variables that characterized the accelerated anaerobic digestion of the grape pomace are: grape pomace ethylic alcohol content, %; carbonylic compounds content, %; fixed residue at 100±5°C, %; fixed residue at 800°C, %; organic carbon content, %; carbon dioxide content, %; grape pomace volume variation, %.

The influence interpretation of the independent variables was accomplished by particularization of the general regression equation:

$$Y = b_o \pm b_i x_i \pm b_{ij} x_i x_j \pm b_{ii} x_{ii}$$

The regression equations particularized for the dependent variable of the grape pomace anaerobic accelerated digestion process are presented in table 3.

The alcohol content is directly influenced by the grape pomace total sugar content and $(NH_4)_3PO_4$ addition (figures 1 and 2). The obtained values prove that the biodegradation process takes place (figures 3 and 4). But the grape pomace biodegradation process is limited by these two factors (total sugar content and $(NH_4)_3PO_4$ addition, especially in the codified values segment of [-2, -1].

**Table 3.** Regression equations for the dependent variables of the grape pomace anaerobic accelerated digestion process

| Regression equations for the dependent variables of the process, $y_i$ | |
|---|---|
| Dependent variables, ($Y_i$) (%) | Regression equation |
| $Y_1$ – ethylic alcohol | $17+1.13X_1+0.82X_2+0.15X_3+0.22X_4+1.22X_1X_2-0.15X_2^2$ |
| $Y_2$ – carbonylic compounds | $19+1.22X_1+1.34X_2+0.15X_3+0.72X_4+0.94X_1X_2+1.13X_2^2$ |
| $Y_3$ – fixed residue at 100±5°C | $29-0.56X_1-0.43X_2+0.12X_3+0.15X_4+1.22X_1X_2+0.22X_2X_3+0.76X_2^2$ |
| $Y_4$ – fixed residue at 800°C | $7.8+1.38X_1-0.46X_2+0.12X_3-0.42X_4-0.79X_1X_2+0.28X_2X_3$ |
| $Y_5$ – organic carbon | $800+1.31X_1+0.86X_2+0.15X_3-0.12X_4-0.93X_1X_2-0.72X_2X_3-0.51X_2^2$ |
| $Y_6$ – carbon dioxide | $46.2-1.14X_1+1.12X_2+0.13X_3-0.96X_1X_2+0.13X_1^2-1.13X_2^2$ |
| $Y_7$ – volume variation | $7-0.22X_1+1.74X_2+0.56X_3-0.24X_4-1.02X_1X_2+0.23X_2X_3-1.15X_1^2+1.22X_2^2$ |

The direct effect of the $(NH_4)_3PO_4$ addition increase is the modification of the grape pomace chemical composition (figures 5, 6 and 7). For example, a $(NH_4)_3PO_4$ addition between 2-2.5% leads to a 12.7% fixed residue content.

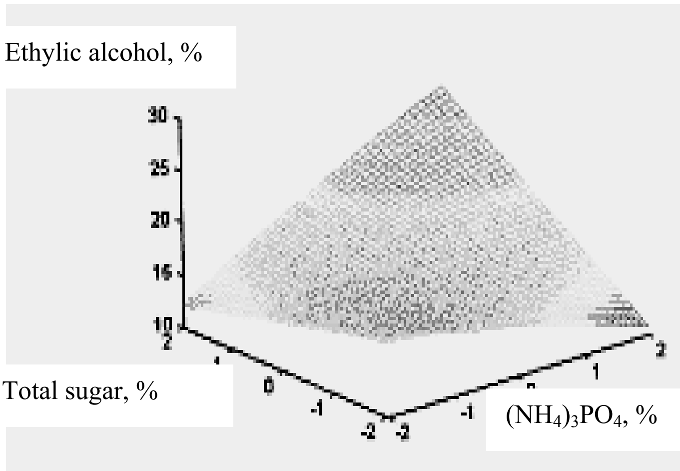


**Figure 1.** The ethylic alcohol variation function of total sugar content and $(NH_4)_3PO_4$ content

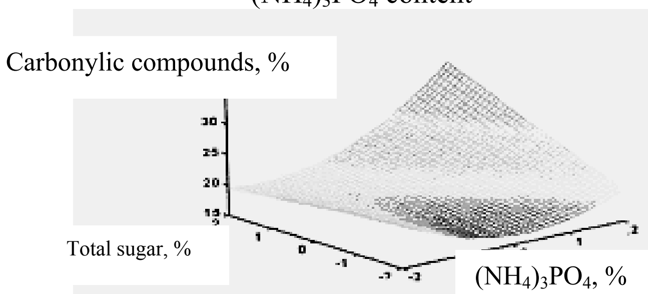


**Figure 2.** The carbonylic compounds variation function of total sugar content and $(NH_4)_3PO_4$ content

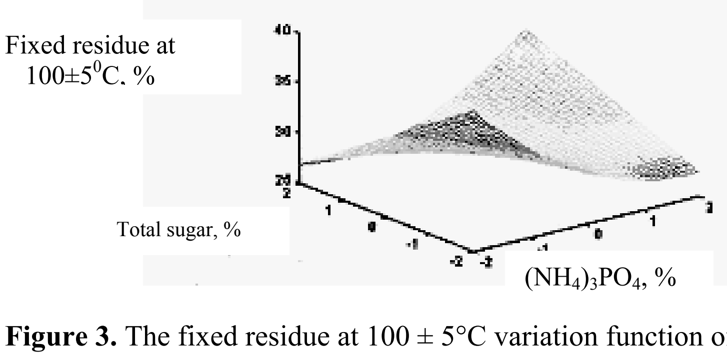


**Figure 3.** The fixed residue at 100 ± 5°C variation function of total sugar content and $(NH_4)_3PO_4$ content

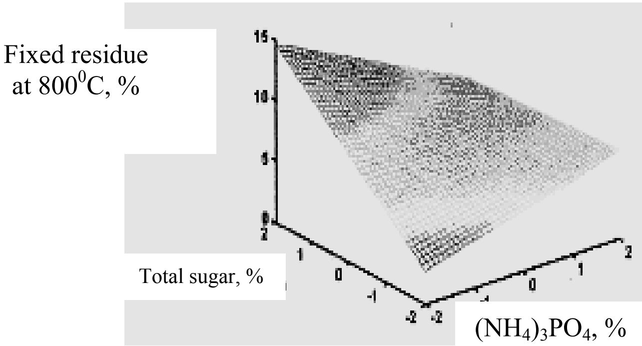


**Figure 4.** The fixed residue at 800°C variation function of total sugar content and $(NH_4)_3PO_4$ content

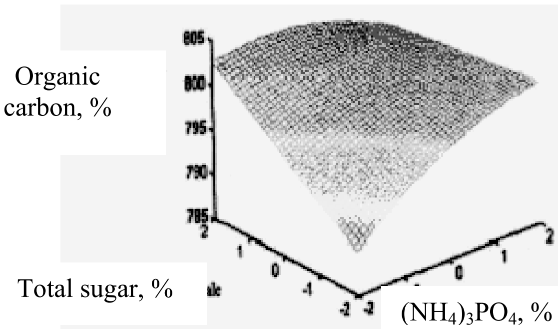


**Figure 5.** The organic carbon variation function of total sugar content and $(NH_4)_3PO_4$ addition

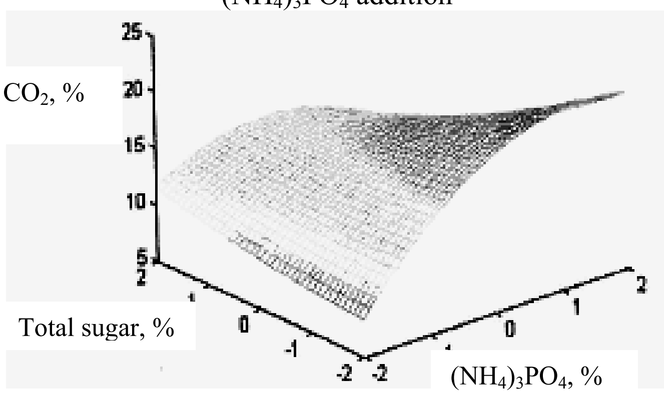


**Figure 6.** The carbon dioxide variation function of total sugar content and $(NH_4)_3PO_4$ addition

If the independent considered parameters were the grape pomace organic composition and $(NH_4)_3PO_4$ addition, the C/N proportion can be measured. At the end of the process, the value of C/N proportion is 17.8-18.2. These values allow the utilization of the fermented grape pomace as animal feed or fertilizer.

In the constant humidity conditions, the $CO_2$ content and grape pomace volume decrease tougher with the decreasing of the organic compound content.

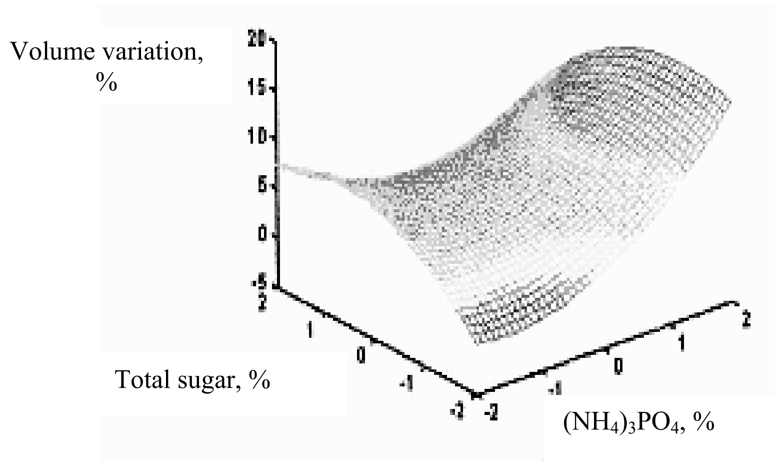


**Figure 7.** The grape pomace volume variation function of total sugar content and $(NH_4)_3PO_4$ addition

The grape pomace final chemical composition, determined at the end of the biodegradation process, is presented in table 4.

**Table 4.** Grape pomace chemical composition at the end of the biodegradation process

| Grape pomace chemical composition at the end of the biodegration process | | | | | |
|---|---|---|---|---|---|
| | Chemical composition reported at the dry matter content[%] | | | | |
| | N | $P_2O_5$ | $K_2O$ | $CaCO_3$ | pH |
| Samples taken during October – January, at the beginning of the biodegradation process | 1.07 | 1.00 | 0.65 | 6.02 | 6.6 |
| Samples taken during February – March, at the ending of the biodegradation process | 1.44 | 1.83 | 0.88 | 8.9 | 6.9 |

## Conclusions

Outcome this experimental research one can come to the following conclusions: the grape pomace anaerobic biodegradation process can be accelerated if we used $(NH_4)_3PO_4$ between 2-3% reported at the total mixture quantity; 90% of the fermenting sugar content was reduce to ethylic alcohol during the grape pomace anaerobic accelerated biodegradation process; the chemical composition of the final product varies function of the biodegradation time; supplying with 2.0-2.5% $(NH_4)_3PO_4$, 98% concentration, has lead reducing of biodegradation time with 32%; anaerobic digestion process inside carbohydrates biodegradation from grape pomace may be augmented with $(NH_4)_3PO_4$ as process accelerator**.**

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