

## BIOETHANOL CONGRUENT DEHYDRATATION

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**Abstract.** Bioethanol is being dehydrated in a number of techniques, but in the production currently the molecular sieves method dominates. It is based on zeolites or on manufacture of these basic materials, water-attracting capacity usage. Since that water adsorbent ability to work is being limited, they are used over azeotropic alcohol concentrations (above 95.5 % mass, or 97.2 % by volume). To the Azeotropic point close is the alcohol purification method. Consequently, in the production of bio-ethanol dehydration alcohol is first being rectified and then the remaining water is separated by a molecular sieve [1]. The molecular sieve technology analysis led us to the thought to combine the rectification and water adsorption processes so that they happen to be in one place and at the same time in one rectifying column. That extraction method of bioethanol could be termed as a “congruent (connected) bioethanol dehydration method”. To verify the congruent method and the assessment in 2008 there were laboratory tests conducted on which the results showed the adsorption of water purification and matching both the usefulness of the technology and energy-saving point of view. The estimation can be concluded that the potential energy savings using the method of dehydration of bioethanol, using congruent process, could save energy up to 70 %.

**Keywords:** bioethanol, congruent, dehydration.

### Equipment and materials for the experiments

#### 1. Congruent rectification and water adsorption equipment

This equipment was made of two Kjeldahl flasks, two flask heaters and two coolers. One of the flasks was filled by spirit of different concentration and by its boiling feeding vapor formed.

When dividing this vapor in two parts, one part was carried into the rectification-adsorption flask, where there was adsorbent pulp (contact mass), but the other part was condensed through the cooler in order to control feeding vapor concentration carried into the contact-flask.

Feeding vapor in the contact-flask by using barbotage tube was carried into the lower layer of the contact mass.

Vapor passing through the contact-mass was transferred to the other cooler and was condensed. When determining the spirit concentration of the condensate, it could be compared with the concentration of the infused vapor and determined both, rectification and water adsorption, the influence caused by the processes.

For determination of the spirit concentration a densimeter set and the table of density-spirit concentration was used taking into consideration temperature correction. Temperature was determined by distance thermometer, more infrequently by glass thermometers.

#### 2. Separation and regeneration equipment of the worked off adsorbent

When completing rectification – adsorption experiment, worked off adsorbent pulp in the residue solution (mainly in the luterwater) was left in the flask. The worked off adsorbent was separated from the residue solution by filtrating it through filter paper of average density using the Bihner filter system (with vacuum).

Filtrated off worked off adsorbent was moved into a metal container, which was put on a burning electric stove, at the temperature of about 400 degrees. Heating was continued till constant mass.

#### 3. Water adsorbents

Powdery laboratory class zeolite and pelleted synthetic material on zeolite base Sylobead MS 564 from the company GRACE Davison in Germany used in production of bioethanol (Jaunpagasts plus, Ltd.) were used as water adsorbents. Sylobead material was used for the first time; granules in the masher were grinded into powder bolting them through a strainer with 0.5 mm mesh.

#### 4. Raw materials containing spirit

As spirit containing raw materials rectification residue product – *EAF* and bioethanol produced by „Jaunpagasts plus” ltd. are used.

#### Results of the experiments

Carrying out several tens of experiments, using as adsorbent both powdery zeolite and grinded Sylobead material, the obtained results on the whole were similar and complied with the theoretically expected ones. One experiment is chosen as an example, when zeolite is used.

The data of the experiment are used for the construction of the diagram shown in the Fig. 1. In the figure curve A shows the changes of the feeding vapor concentration during the experiment, but the curve B – at the same time, changes of dehydrated spirit vapor concentration by rectification and water adsorption.

It is obvious that in the above azeotropic concentrations (above 97.2 % vol.) where only water adsorption takes place, increase of the spirit concentration is convincing – from 0.3 % at 99.7 % infused feeding vapor concentration up to 1.5 % at 97.6 %.

Favorable influence of adsorbent maintained also in below-azeotropic concentrations. It is obvious from the position of the curve C in the figure. The curve C is constructed from the table of balance data [2] which shows coherence between boiling spirit concentration and concentration distributed from its vapor. Consequently the position of this curve shows that the experiment dealt with here, for example, at the feeding vapor concentration 99 % vol. as a result of rectification process concentration increased for about 2.5 %, but in addition, as a result of water adsorption, it increased for about one more per cent, totally about 3.7 %. It is essentially important at concentrations above 94% vol., that by rectification only a slight reinforcement effect can be reached, which by increase of concentration, gradually decreases and at 97.2 % vol. is not possible at all. Therefore, in order to obtain spirit of high concentration, in rectification columns a lot (even up to 100) of reinforcement plates are inserted. To reach the highest concentrations also increased amount of heat has to be used. In the literature [3], for example, it is depicted that for increase of the spirit concentration with rectification from 96.0 till 96.5 % vol., use of vapor increases for 66 % (from 15 kg div<sup>-1</sup> till 25 kg div<sup>-1</sup>). Therefore supplementing rectification with water adsorption can give remarkable economy of energy.

Fig. 1 shows that in the experiment dealt with, binding ability of adsorbent water used is exhausted at spirit concentration of about 77 %. In other experiments, when greater amount of adsorbent was used, water binding continued also in lower concentrations.

In order to determine the proportion of separated water in both dehydration processes during the whole course of the experiment, steps are drawn in the diagram, beginning with the spirit concentration, where adsorption process had already finished. From the verticals drawn in steps, where they cross with the curve C, the proportions of separated water amount during both processes can be read. The results are shown in Table 1.

Table 1

**Process proportions of rectification and water adsorption at different spirit vapor concentrations**

Infused vapor conc., %		Dehydrated vapor conc., %		Difference, tot. %mas	Difference attained by rectification		Difference attained by adsorption	
vol	mas	vol	mas		%m	part of tot. diff.	%m	part of tot. diff.
77.0	70.1	87.7	82.6	12.5	12.5	1.00	0.0	0.00
87.7	82.6	93.2	90.0	7.4	5.7	0.77	1.7	0.23
93.2	90.0	95.9	93.7	3.7	1.1	0.31	2.6	0.69
95.9	93.7	98.6	96.4	2.7	0.3	0.12	2.4	0.88
98.6	96.4	100	100	3.6	0.0	0.00	3.6	1.00
Total				30.0	19.6		10.4	

From the data of Table 1 we can conclude that in this experiment by rectification  $(19.6:30.0) \cdot 100 = 65\%$ , but by adsorption  $(10.4:30.0) \cdot 100 = 34\%$  of water are separated.

In the diagram between the curves A and B the number of the steps drawn show the number of reinforcement steps necessary for dehydration, resp. the theoretical number of plates to be inserted into the rectification column.

In this experiment the fixed number of reinforcement steps is five.

Potential economy of energy in the technological point of bioethanol dehydration by applying congruous method might be even up to 70%.

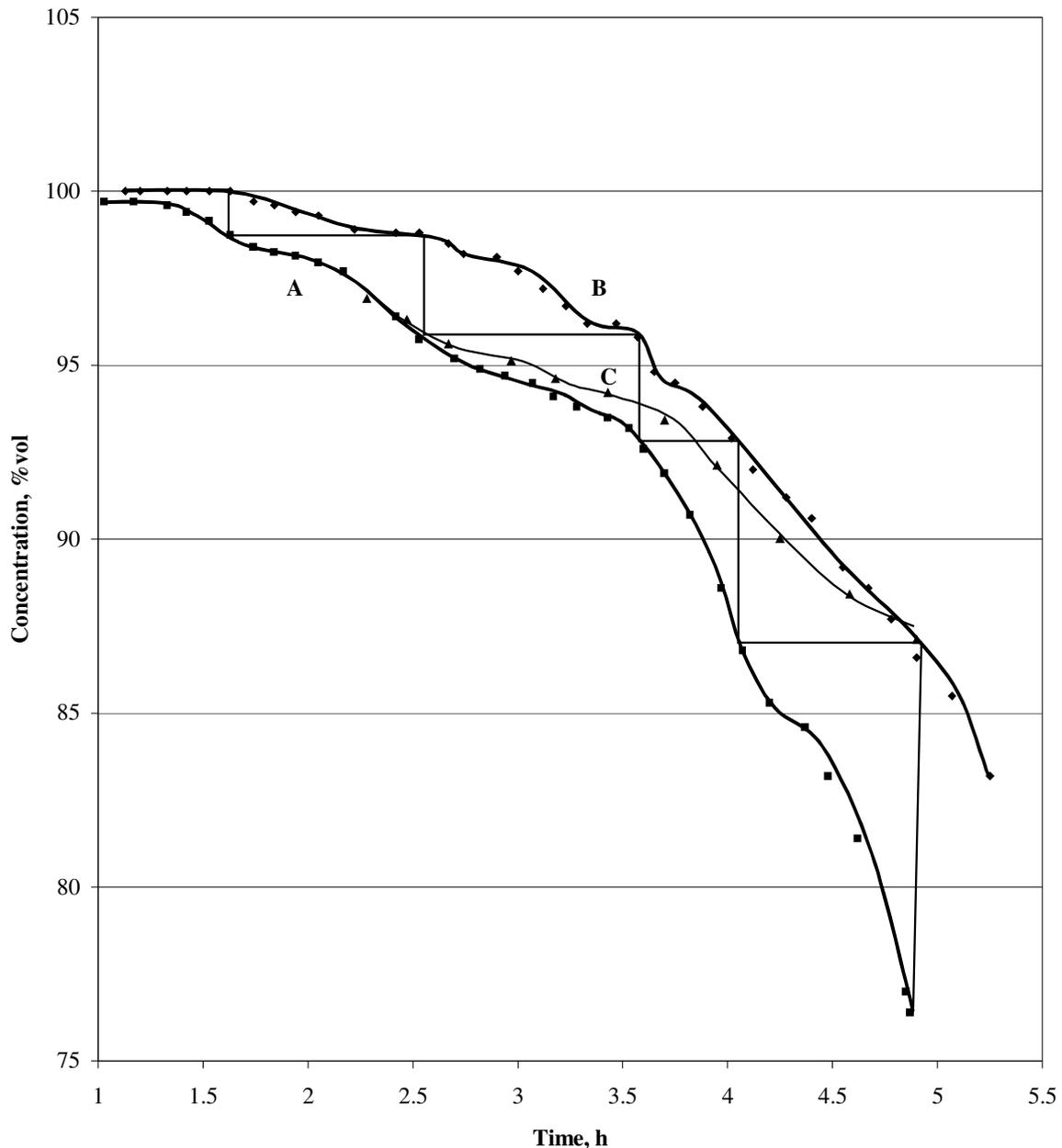


Fig. 1. **Diagram of congruous dehydration process:** A – changes of feeding conc. % vol.; B – changes of distillate conc. % vol.; C – changes of balance conc. % vol

### Conclusions

1. Instead of the divided bioethanol dehydration method, when till the azeotropic concentration it is carried out by rectification and further water adsorption, it is preferable to combine together both processes in the rectification equipment, thus obtaining technological and economical preferences.

2. Potentiality of combining both dehydration processes in the laboratory equipment was fully proved during the carried out experiments bringing bioethanol till the condition of absolute spirit.
3. The most significant technological preferences of congruent bioethanol dehydration is a considerably reduced number of reinforcement steps (plates) and safer regeneration of adsorbent because instead of spirit water is separated from the worked off adsorbent.
4. The economic effect of the congruent dehydration of bioethanol is up to 70 % of heat energy economy.

### References

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