

## **EXPLORATION OF TECHNOLOGICAL EQUIPMENT FOR A NEW BIOETHANOL DEHYDRATION METHOD**

### **NAUJO BIOETANOLIO DEHIDRATACIJOS METODO TECHNOLOGINĒS ĪRANGOS TYRIMAI**

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In conformity with the requirements of the EU directives, Latvia has to increase the annual utilisation of biofuel, including bioethanol, in transport. In order to decrease the consumption of energy for the production of bioethanol, a new bioethanol dehydration method has been developed at the Latvia University of Agriculture (LUA), for which an application has been submitted for the European Patent No.EP2316549 "*Method and device for removing water from ethanol by combined adsorption and distillation*". In order to carry out testing of the structural units of the developed technology to achieve optimum solutions, in cooperation with the Institute of Physical Energetics experimental equipment for testing the variants of the bioethanol dehydration technology has been created within the framework of the European Regional Development Fund project "*Innovative bioethanol dehydration technologies and elaboration of the measuring equipment for the determination of its parameters*" No.2010/0281/2DP/2.1.1.1.0/10/APIA/VIAA/003 at the Research Institute of Agricultural Machinery, Latvia University of Agriculture.

Considering the fact that a patent EP 2524722 A1 for the developed experimental equipment has been submitted for the European Patent Office, entitled "Device for semi-dry congruent dehydration of bioethanol". Application published in the European Patent Office the official Bulletin 2012/47.

Therefore the equipment and its units which are necessary for the implementation of the technology of the method are shown in the form of block schemes. The research results include also the results of the already conducted studies of the dosing equipment for the adsorbent granules, as well as the consequent conclusions.

*Bioethanol dehydration, dosing equipment for the adsorbent granules, a rectification column.*

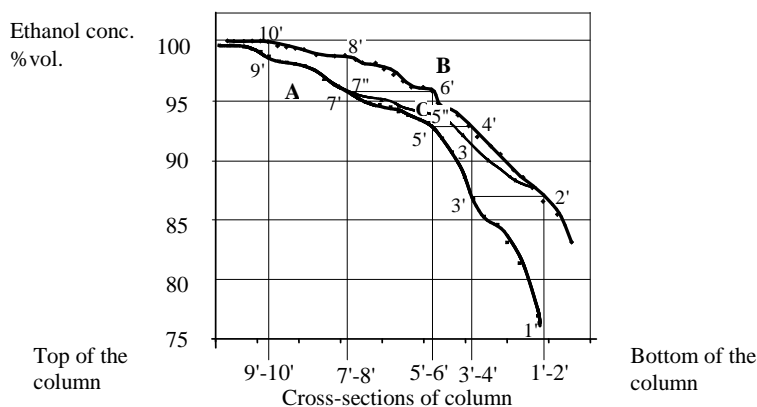
### **Introduction**

A dehydration technology, mostly used at present in the production of bioethanol, is such a dehydration technology of the leaven distillate when, first of all,

96.5% concentration of alcohol by volume is obtained in the conventional rectification columns and, after that, more complete extraction of rectified alcohol from water is carried out by molecular grids (Lyons et al., 1995).

In contrast to that technology, the Latvia University of Agriculture (LUA) has developed a new bioethanol dehydration method according to which, applying the bioethanol semi-dry congruent dehydration principle, both stages are joined together forming a new production technology. The principles of the bioethanol semi-dry congruent dehydration technology and equipment will be applied for a European patent. (European patent Nr. EP2316549)

The bioethanol semi-dry congruent dehydration principle determines that separation of water from alcohol takes place by means of simultaneous water adsorption and rectification in a downflowing layer of loose moist adsorbent granules. The bulk of granules is constantly restored by adding fresh, active granules in the top which, moving down, gradually become saturated with water, absorbing it from the dehydrated alcohol vapour in the middle and removing the used, water saturated granules from the bottom. In the cross sections where the alcohol concentration of the liquid phase is below 97.2% vol. (azeotropic concentration), mass and heat exchange takes place on the surface of the moving layer of granules which ensures implementation of the rectification process just as it proceeds in the filling-type\_rectification columns (Stabnikov, 1969). Application of the bioethanol semi-dry congruent dehydration principle is depicted in Figure 1.



**Fig. 1.** Diagram of a congruent dehydration process: A - changes in the feeding conc. % vol.; B - changes in the distillate conc. % vol.; C - changes in balance conc. % vol

**1 pav.** Kongruentīnēs dehidratācijas eigos diagrama: A – maitinimo koncentrācijas pokyčiai; B – distiliato koncentrācijas pokyčiai, % talp; C – pusiausvros koncentrācijas pokyčiai % talp

As example how the bioethanol semi-dry congruent dehydration principle is implemented and how either of the water separation processes affects increase in the total alcohol concentration in various cross sections of Block 1 is shown in Figure 1. (Bremers et al., 2010). The diagram is created on the basis of the relation-

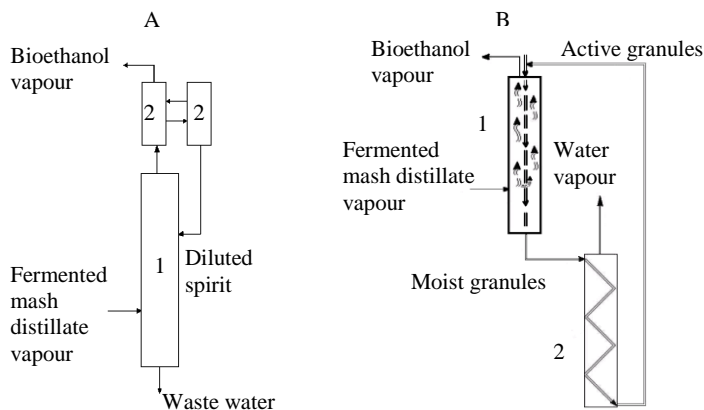
ships established in the inventors' experiments between the concentration of liquid boiling alcohol and the concentration of vapour obtained from it in the presence of a water adsorbent.

By marking on Curve A the concentration of liquid boiling alcohol in a certain cross section of Block 1, the vertically connected point on Curve B shows the concentration of the alcohol vapour extracted from this liquid. (Bremers et al., 2011)

Curve C is drawn using the data from the alcohol solution-vapour equilibrium table (Stabnikov, 1969), and it theoretically depicts the concentration correlations of both these phases only in the rectification process.

In the example shown in Figure 1 in the cross section 1'-2' of Block 1 where there is a boiling alcohol solution of about 77% concentration, only the rectification process is fulfilled, and alcohol vapour emanates from this solution with a concentration of about 87%. In the cross section 3'-4', there proceed already both water separation processes. The alcohol concentration rising from 87% to 93% as a result of the two processes, by approximately 5% relate to rectification (3'-3''), and only 1% is due to adsorption (3''-4'). In the cross section 7'-8', on the contrary, the impact of rectification is negligible, and almost all the increase in the concentration of the alcohol solution arises from the water adsorption procedure (the cross section 7''-8''). In the above azeotropic concentrations of alcohol (above 97.2% vol.), only water adsorption takes place (for instance, in the cross section 9'-10'). (Rucins et al., 2012)

A comparison of both the technologies mentioned above is shown in Figure 2 in the form of block schemes.



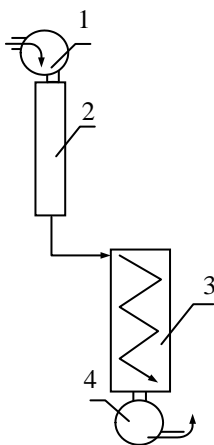
**Fig. 2.** Comparison of the dehydration technologies (European Patent No. EP2316549): A - the present technology: 1 - the rectification column, 2 - molecular grids; B - the recommended technology: 1 - the dehydration column, 2 - the granule regenerator

**2 pav.** Dehidracijos technologijų palyginimas (EI patentas Nr. EP2316549): A – Dabartinė technologija: 1 – rektifikacijos kolona, 2 – molekuliniai sietai; B – Rekomenduojama technologija: 1 – dehidracijos kolona, 2 – granuliu regeneratorius

## Object and methods

Experimental equipment for dosing and sealing the water adsorbent granules was designed and made for technical pre-research of the developed and patented congruent bioethanol dehydration technology intended for the implementation of the technological process and testing its structural units. As one of the issues to be solved, was to carry out designing the dosing and sealing units of the water adsorbent granules for the experimental equipment in order to conduct investigations about its compliance with the requirements of the technological process.

In the equipment these units are placed on the spots where the adsorbent granules are introduced and removed. They are presented as block schemes in Figure 3. By means of these units the intensity of the granule motion is varied with simultaneous blocking the alcohol vapour escape from the equipment.



**Fig. 3.** A technological block scheme of the pilot equipment with the sealing and dosing units: 1 - the sealing unit of the equipment; 2 - the bioethanol dehydration column; 3 - the granule regenerator; 4 - the granule dosing unit

**3 pav.** Modelinio įrenginio technologinė blokinė schema su įrenginio hermetizavimo ir dozavimo mazgais: 1 – įrenginio hermetizavimo mazgas; 2 – bioetanolio dehidracijos kolona; 3 – granulių regeneratorium; 4 – granulių dozavimo mazgas

In the variant studied these tasks were fulfilled using a granule dosing and sealing unit with a spherical core, providing it with a transverse wall fixed in the channel of the doser (Figure 4). When rotational movement was applied to the doser core, in position 2 the granules fell into the chamber but in position 3 they fell out of the chamber.

In the tests of the granule dosing and sealing units as a water adsorbent was used Sylobead MS 564 - a granular synthetic material based on celoite, which is applied in the production of bioethanol.



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3

**Fig. 4.** The granule dosing and sealing unit with a spherical core  
**4 pav.** Granulių dozavimo hermetizavimo mazgas su rutulio pavidalo šerdimi

Experiments were made in order to determine variations in the size of the particles by repeated passing the granules through the dosing and sealing unit. The total number of passings of the granules was ten. After that the granules were sifted through screens of different sizes with the mesh dimensions from 0.56 mm to 2.5 mm and weighed by the sizes of the particles.

### Results and discussions

The granule dosing and sealing units must ensure regulation of the throughput of the water adsorbent granules in the equipment with simultaneous prevention of the escape of alcohol vapour into the surrounding environment. The results of the accomplished studies confirmed the fact that, using a granule dosing and sealing unit with a spherical core and varying the revolutions of the core it is possible to change the intensity of the granule motion in the equipment. When the revolutions of the doser were increased, the throughput ability of the doser increased (see Table 1). However, after multiple passing of the granules through the doser, an undesirable by-effect was observed – splitting of the granules.

**Table 1.** The throughput ability of the dosing and sealing unit of the adsorbent granules depending on the revolutions of its core

**1 lentelė.** Adsorbento granulių dozavimo hermetizavimo mazgo pralaidumas, priklausomai nuo jo šerdies apsisukimų skaičiaus

Revolutions of the doser core $\text{min}^{-1}$	Throughput of the doser, $\text{kg h}^{-1}$
16	11.8
5	3.9
4	3.2

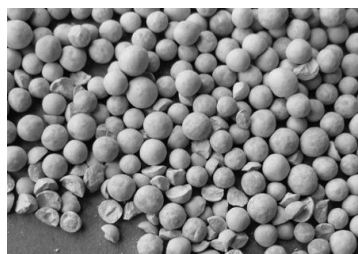
Splitting of the granules takes place in positions 2 and 3 of the spherical core of the dosing and sealing unit (Fig. 4). Variations in the size of the granule fractions after their multiple passing through the doser are shown in Table 2.

**Table 2.** Variations in the size of the granule fractions after their multiple passing through the doser.

**2 lentelė.** Adsorbento granuliu dydžių sudėties pokyčiai, jas daug kartų praleidžiant pro dozatorių.

Limits of the granule sizes, mm	Amount of the granule fractions		Variations in the granule fractions related to one cycle, %
	before the experiments, %	after 10 cycles, %	
2...2.5	54.0	28.0	-2.6
1...2	46.0	66.0	+2.0
0.56...1	0	5.0	+0.5
below 0.56	0	2.0	+0.2
	100	100	

Variations in the form of the granule particles after their passing through the doser, in comparison with the initial spherical form, are evident in Figure 7. The pouring capacity of the split balls shown in the picture is worse than that of the initial round ones; therefore there is a risk of possible plugging of the granule flow in the equipment, which is not allowed.



**Fig. 7.** Variations in the form of the granule particles after their passing through the doser

**7 pav.** Adsorbento granuliu forma, jas daug kartų praleidžiant pro dozatorių

### Conclusion

1. It has been found out in the investigations that, using the granule dosing and sealing unit with a spherical core and varying the revolutions of its core it is possible to change the intensity of the granule motion in the equipment.
2. Using the granule dosing and sealing unit with a spherical core, an undesirable by-effect was observed after multiple passing of the granules through the doser – splitting of the granules.
3. After multiple passing of the granules through the dosing and sealing unit with a spherical core the amount of granules with the dimensions of 2...2.5 mm decreased 2 times with a corresponding increase in the amount of the split granules.
4. Results of the research show that the granules dosing unit with a spherical core sealing in this equipment do not acceptable because splitting of the granules grinding is not permitted. It is necessary to carry out additional studies for finding optimal solution granules dosing and sealing units.

## Acknowledgements

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7. An application for the European patent EP 2524722 A1 for the developed experimental equipment has been submitted for the European Patent Office, entitled "Device for semi-dry congruent dehydration of bioethanol". *Application published in the European Patent Office the official Bulletin* 2012/47.

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## NAUJO BIOETANOLIO DEHIDRATACIJOS METODO TECHNOLOGINĒS ĪRANGOS TYRIMAI

### Santrauka

Vadovaudamasi ES direktyvos reikalavimais, Latvija kasmet turi didinti biokuro, įskaitant bioetanolį, naudojimą transporto reikmėms. Siekiant sumažinti energijos sąnaudas bioetanolio gamyboje, Latvijos Žemės ūkio universitete yra parengtas naujas bioetanolio dehidratacijos metodas, kuris užregistruotas el. paten-

tui įgyti. Bioetanolio dehidratacijos technologijos variantams patikrinti Latvijos Žemės ūkio universiteto agentūros Žemės ūkio technikos mokslo institute, bendradarbiaujant su Fizikinės energetikos institutu pagal ERPF projektą „*Inovacinės bioetanolio dehidratacijos technologijos ir jo parametrų nustatymo* matavimo prietaisų rengimas“ Nr.2010/0281/2DP/2.1.1.1.0/10/APIA/VIAA/003 sukurta eksperimentinė įranga, kad būtų galima atlikti parengtos technologijos konstrukcinių mazgų patikrą optimaliems sprendimams pasiekti.

Atsižvelgiant į faktą, kad parengtai eksperimentinei įrangai skirtas ir registruotas patentas Nr. LV 110068 LV Patentų valdyboje „Bioetanolio pusiau sausos kongruentinės dehidratacijos įrangos adsorbento granuliu regeneracinis blokas“, kuris yra prioritetinis dokumentas Europos patento paraiškai Nr.EP2524722 A1, *Kompaktinis bioetanolio pusiau sausos kongruentinės dehidratacijos įrangos komplektas*, todėl metodo technologijai realizuoti būtina įranga ir jos mazgai parodyti blokinių schemų forma. Tyrimų rezultatuose parodyti atliktų adsorbento granuliu dozavimo įrangos tyrimų rezultatai, taip pat su jais susijusios išvados.

*Bioetanolio dehidratacija, adsorbento granuliu dozavimo įranga, rektifikacijos kolona.*

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## ИССЛЕДОВАНИЕ ТЕХНОЛОГИЧЕСКОГО ОБОРУДОВАНИЯ ДЛЯ НОВОГО МЕТОДА ДЕГИДРАТАЦИЯ БИОЭТАНОЛА

### Аннотация

Требования директив ЕС к Латвии предусматривают необходимость ежегодного повышения использования биотоплива и, в том числе, биоэтанола. Для уменьшения расхода энергии при производстве биоэтанола в Латвийском сельскохозяйственном университете разработан новый метод дегидратации, на который подана заявка на получение Е-патента. Для проверки вариантов дегидратации в рамках проекта ЕС «Разработка инновационной технологии дегидратации биоэтанола и измерительной аппаратуры для определения ее параметров» Nr.2010/0281/2DP/2.1.1.1.0/10/ APIA/VIAA/003 создано экспериментальное оборудование для выявления оптимальных параметров технологии.

Поскольку на экспериментальное оборудование получен патент Латвии LV 110068, который является приоритетным документом патентной заявки Nr.EP2524722 A1 «Компактный комплект оборудования для полусухой конгруэнтной дегидратации биоэтанола», то необходимое оборудование и узлы показаны в форме блок-схемы. В работе отражены результаты исследований оборудования для дозировки гранул адсорбента и сделаны соответствующие выводы.

*Дегидратация биоэтанола, оборудование для дозирования гранул адсорбента, реktификационная колонна.*