

ADAPTATION OF TRANSESTERIFICATION REACTION CONDITIONS FOR THE INDUSTRIAL SYNTHESIS OF LACTIC ACID ESTERS WITH SELECTED ALCOHOLS

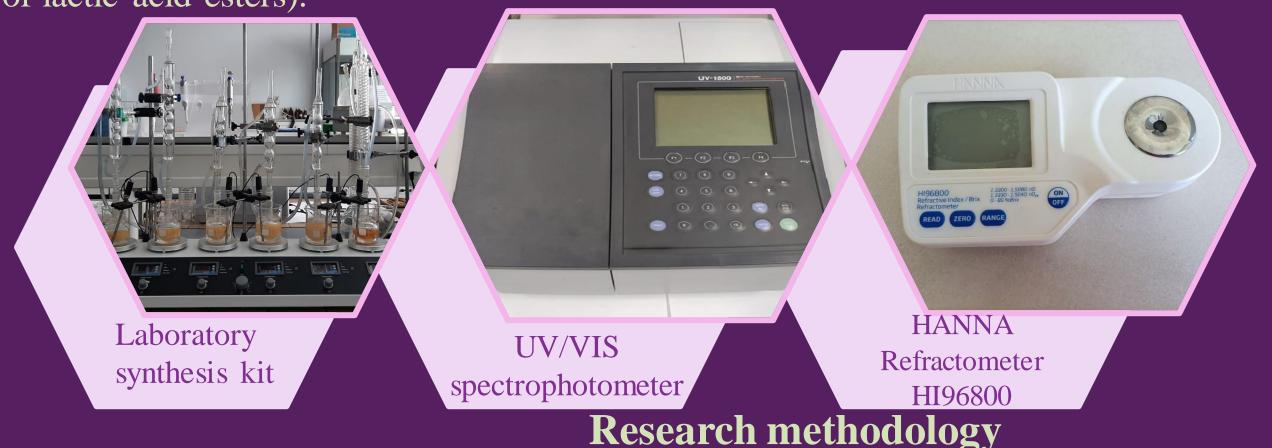
PRZEMYSŁAW RUTANA, AGATA POPIS, MATEUSZ KAJKOWSKI, JULIA SULIŃSKA, ANETA NOWAKOWSKA, MARZENA KOWALEWSKA, DANIEL FIJAŁKOWSKI, MICHAŁ DOŁĘBSKI, BARTŁOMIEJ SULIŃSKI, ADAM GAWLIK, PIOTR GRABAREK¹

¹WARSAW UNIVERSITY OF TECHNOLOGY, FACULTY OF CIVIL ENGINEERING, MECHANICS AND PETROCHEMISTRY, ŁUKASIEWICZA 17, 09-400, PŁOCK.



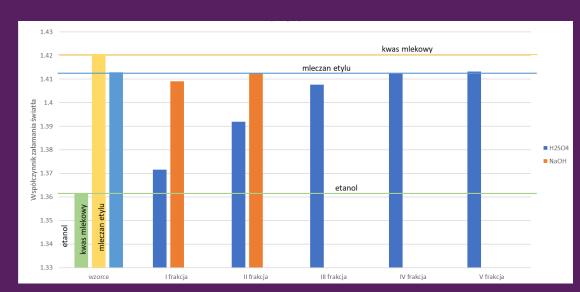
Introduction

Lactic acid esters are gaining popularity as petrochemicals that can find applications in many industries. They are used in the food industry, where they are used as flavourings and preservatives. Further opportunities for their use are in the pharmaceutical and cosmetic industries, where lactates are used as solvents and additives. Lactic acid esters are characterised by relatively high prices. This is due to the renewable origin of many of the raw materials for their production. In addition, separation and purification processes for the synthesised esters are characterised by high costs. Consequently, upgrades in the synthesis and purification methods of the products obtained are necessary. Lactic acid esters are most often obtained by the esterification reaction of alcohols with lactic acid. During the development of the grant topic, similarities between the synthesis of lactates and the acid transesterification of vegetable oils were analysed. Many common factors were observed, including reaction parameters, and PNKCh members will therefore attempt to apply transesterification conditions to the preparation of lactic acid esters).



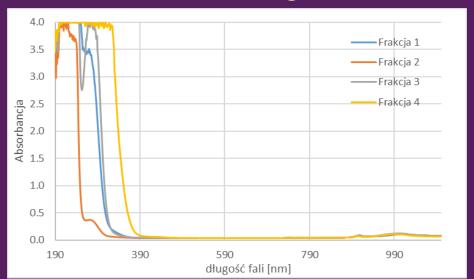
Purpose of research

The aim of this year's grant was to investigate the adaptability of transesterification technology for the synthesis of selected lactid acid esters.

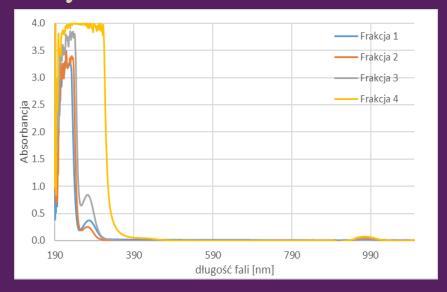


UV/VIS spectrum of the reference solutions

UV-VIS spectra for the reaction of reagents



Example spectrum obtained for a sample obtained using NaOH as catalyst with ethanol in a 1:2 ratio



Example spectrum obtained for a sample obtained using H₂SO₄ as catalyst with ethanol 99,8% in a 1:2 ratio

SYNTHESIS

Lactic acid and ethanol were mixed in different ratios and a catalyst was used. Atmospheric distillation was then used to separate the sample components. After the distillation of the last fraction (lactate) had started and several cm³ of ester had been collected, the mixture began to darken and became charred. New samples were then prepared and the mixture was separated by vacuum distillation. The lactate fraction was collected.

UV/VIS SPECTROSCOPY

The previously obtained samples were examined using a UV/VIS spectrophotometer with a resolution capacity of 1 nm and a measurement range of 190 - 1100 nm. The test was carried out in 5 nm quartz cuvettes. The test solution was introduced into the cuvette and the spectrophotometer was started. The presence of lactic acid esters was checked by comparing the peak of the standard (330 nm) and the test samples. Peaks corresponding to the esters were observed on the spectrum obtained.

REFRACTIVE INDEX OF LIGHT

On the basis of the results for the first series of mixtures tested, in which technical (un-esterified) ethanol was used for synthesis, none of the distillates had a refractive index of 1.4128, characteristic of ethyl lactate. Due to the possibility of adverse effects of substrate un-fertilisation and water formation as a result of the esterification reaction, esterification in the presence of a drying agent in the form of SilicaGel was attempted. The use of the drying agent produced significantly higher refractive indices, indicating a shift in the reaction equilibrium towards the production of esters. The main objective of the Rector's grant was to investigate the possibility of adapting the transesterification conditions of vegetable oils to the synthesis of lactic acid esters - lactates.

Conclusion

The main objective of the Rector's grant carried out by students of the Płock Scientific Circle of Chemists was to investigate the possibility of adapting the transesterification conditions of vegetable oils for the synthesis of lactic acid esters - lactates. On the basis of the completed first part of the work, positive results of such an attempt can be observed. The synthesis proceeds with good yields as confirmed by the absence of lactic acid in the samples tested. According to the reaction variants used, dehydrating agents or anhydrous substrates should be used in the reaction. A problematic part of the research work was the separation of the reaction mixture. During the course of the Rector's grant, it became apparent that distillation at atmospheric pressure was not a good separation method for this type of mixture. This may have seemed obvious due to the presence of catalysts that do not leach out after the reaction. The lack of this unit operation was due to the attempt to bypass this step, which in industry would have resulted in large amounts of effluent and the need to manage it. Consequently, an attempt was made to distil first unreacted ethanol and eventually ethyl lactate by vacuum distillation. This proved to be a valid approach. During separation of the reaction mixture by this method, the distillation residue was not charred and could be recycled back into the process.

Międzynarodowe Sympozjum im. Bolesława Krzysztofika ΜΩUA