ABSTRACT

The aim of present thesis was to discover ionic liquid ability to dissolve lignocellulosic biomass and convert it more valuable products. Work is focused on rice husks and experimental study has performed in order to find the optimized conditions for producing quality fractions from this feedstock. Ionic liquids 1-ethyl-3-methylimidazolium acetate [EMim][OAc], 1-ethyl-3-methyl-imidazolium chloride [BMim][Cl] and triethylammoniumhydrogensulfate [TEA][HSO_4] were investigated as green solvents for this purpose. Dialkylimidazolium salts proved to be effective solvents to dissolve biomass with a longer period (at least 36 h). Different antisolvents as water, alcohols and acetone were tested in regeneration stage, and was found that more suitable are alcohols and water. Dissolving process is affected by temperature, pretreatment time and ratio of biomass and ionic liquid. Further procedure depends on viscosity and desolvents, which were used as regeneration agents. Disadvantage of dialkylimidazolium based ionic liquids is high cost, therefore further experiments were carried out using [TEA][HSO_4].

[TEA][HSO_4] was synthesized according to the neutralization reaction between triethylamine and sulfuric acid. Water content was determined with Karl Fischer. According to literature ionic liquid solution with 20wt% H_2O was used in pretreatment procedure.

Fractionating process called IonoSolv process final products were cellulose enriched pulp and lignin. Rice husks which have high ash and lignin content need to have pretreatment temperature at least 120 ^{0} C and 5 h. Most of the lignin is extracted if higher temperature and longer time are used, but pretreatment parameters should not exceed 150 ^{0} C and 4 hours when delignification decreased due to pseudo-lignin formation. Rice husk biomass contains 39,44% lignin as measured with compositional analysis. Most of the lignin was extracted at 150 ^{0} C 1,5+1,5 h. Hemicellulose was extracted completely at 150 ^{0} C 3 h. Mass loss is higher if higher pretreatment temperatures are used. This due to extractives and hemicellulose and lignin fragments (e.g. furfurals), which accumulate in ionic liquid and are hard to regenerate.

Cellulose content and ash content are not affected by ionoSolv process (difference 3-5%). Final product is silica-enriched cellulose pulp, where glucan content doubles compared with untreated biomass (150 ^{0} C and 2 h). Disadvantage of this process is incomplete lignin removal – 150 ^{0} C and 3 h treated pulp composition was following: 64,16% cellulose, 17,92% ash, 17,91% lignin.

Main advantage of using [TEA][HSO_4] is that process is not water sensitive and there is no need to proceed expensive biomass drying process. Pretreatment of rice husks with [TEA][HSO_4] 20wt% H2O solution was effective to remove most of lignin from biomass. IonoSolv method is energy effective because H_2O, ethanol and ionic liquid are recyclable.

Rice husks are challenging feedstock that is indicated by enzymatic saccharification yield of untreated biomass (8%). Low yield shows that cellulose fraction is well protected of biological attach. Saccharification yield depends on how effective is lignin removal in pretreatment stage. Maximum saccharification yield 38% was received on 150 ^{0} C and 2 h pretreated biomass, but it is considered as low result compared to other biomasses, where saccharification yield could reach more than 75%. Because of that, rice husks are not the best feedstock for producing biofuels and there should be considered other outputs like developing new materials, for example silica-enriched cellulose fiber or aerogel made of lignin. Achieved results can be used to developing new materials.