Final Technical Report

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Summary

The goal of this project is the development of novel catalysts and knowledge of reaction pathways and mechanisms for conversion of biomass-based compounds to fuels that are compatible with oil-based fuels and with acceptable or superior fuel properties. The research scope included both catalysts to convert lignocellulosic biomass-based molecules (from pyrolysis) and vegetable oil-based molecules (i.e., triglycerides and fatty acid methyl esters). This project comprised five technical tasks. Each task is briefly introduced below, and major technical accomplishments summarized. Technical accomplishments were described in greater detail in the quarterly progress reports, and in even more detail in the >50 publications acknowledging this DoE project funding (list of publications and presentations included at the end of this report). The results of this research added greatly to the knowledge base necessary for upgrading of pyrolysis oil to hydrocarbon fuels and chemicals, and for conversion of vegetable oils to fungible diesel fuel. Numerous new catalysts and catalytic reaction systems were developed for upgrading particular compounds or compound families found in the biomass-based pyrolysis oils and vegetable oils. Methods to mitigate catalyst deactivation were investigated, including novel reaction/separation systems. Performance and emission characteristics of biofuels in flames and engines were measured. Importantly, the knowledge developed from this project became the basis for a subsequent collaborative proposal led by our research group, involving researchers from the University of Wisconsin, the University of Pittsburg, and the Idaho National Lab, for the DoE Carbon, Hydrogen and Separations Efficiency (CHASE) program, which was subsequently funded (one of only four projects awarded in the CHASE program). The CHASE project examined novel catalytic processes for lignocellulosic biomass conversion as well as technoeconomic analyses for process options for maximum carbon capture and hydrogen efficiency.

Our research approach combined catalyst synthesis, measurements of catalyst activity and selectivity in different reactor systems and conditions, and detailed catalyst characterization to develop fundamental understanding of reaction pathways and the capability to predict product distributions. Nearly all of the candidate catalysts were prepared in-house via standard techniques such as impregnation, co-impregnation, or chemical vapor deposition. Supports were usually purchased, but in some cases coprecipitation was used to simultaneously create the support and active component, which can be advantageous for strong active component-support interactions and for achieving high active component dispersion. In-house synthesis also allowed for studies of the effects on catalyst activity and selectivity of such factors as support porosity, calcination temperature, and reduction/activation conditions. Depending on the physical characteristics of the molecule, catalyst activity measurements were carried out in tubular flow reactors (for vapor phase reactions) or stirred tank reactors (for liquid phase reactions) over a wide range of pressures and temperatures. Reactant and product concentrations were measured using gas chromatography (both on-line and off-line, with TCD, FID, and/or mass spectrometric detection). For promising catalysts, detailed physicochemical characterization was carried out using FTIR, Raman, XPS, and XRD spectroscopies (all available in our laboratories) and TEM spectroscopy (available at OU). Additional methods included temperature programmed techniques (TPD, TPO) and surface area measurements by nitrogen adsorption techniques.

Task A: Fundamental catalytic studies to develop catalysts for conversion of pyrolysis oil into fungible fuel

Task A focused on conversion of pyrolysis oil molecules whose carbon number is already appropriate for gasoline or diesel fuel, but which are not fungible fuel molecules because of oxygen content. These compounds may be multiply oxygenated, may contain carboxylic oxygen groups, and/or may be heterocyclic compounds with oxygen in the ring – e.g., phenols, furfurals, carboxylic acids, and others.

For task A, studies included supported metal catalysts (primarily Cu, Pd, and Pt metals on silica and alumina supports), bimetallic catalysts (Pt-Sn) on silica and alumina supports, and acidic and cation-exchanged zeolites. Important parameters include metal loading, type of precursor, calcining, reduction and activation conditions, and both type of cation and extent of exchange.

In early studies, the deoxygenation of benzaldehyde, a model compound, to benzene and toluene was investigated on basic CsNaX, and NaX zeolite catalysts (Peralta, 2009). While both benzene and toluene are desirable deoxygenated products, toluene is the more desirable product as it represents a higher capture of carbon. Maximizing carbon capture and minimizing hydrogen consumption were recognized as important objectives for any biofuels process. It was observed that as-prepared CsNaX, containing Cs in excess, displays high activity for direct decarbonylation of benzaldehyde to benzene. However, in parallel with the decarbonylation reaction, condensation of surface products occurs. Therefore, the lower pore volume of catalyst having excess Cs leads to lower catalyst stability. Likewise, catalyst stability was recognized early in the project as an important consideration in catalyst development. Decomposition of surface condensation products results in further evolution of benzene and toluene. It is observed that gas-phase H_2 can play an important role by reducing the residence time of surface intermediates, thus decreasing the amount of condensation products that accumulate and lead to catalyst deactivation. Hydrogen transfer to the condensation surface products accelerates the decomposition of these condensation compounds primarily into toluene.

Transalkylation improves carbon capture during upgrading of methoxylated phenolic species which are common in pyrolysis oils. Transalkylation was studied over several zeolite catalysts including HZSM-5 (Zhu, 2010), Ga-modified ZSM-5 (Ausavasukhi, 2009), and H-beta zeolite. These studies elucidated the role of acid sites for efficient capture of methoxyl groups via transalkylation with metal sites for deoxygenation. Fundamental understanding of the role of hydrogen transfer during conversion of phenolic compounds was gained by study of tetralone conversion over HY zeolite. Knowledge of the mechanisms and catalysts was foundational to further research during the DoE CHASE project, on alkylation of lignin-derived phenolics with small oxygenates generated by torrefaction and pyrolysis of biomass.

Task B: Fundamental catalytic studies to develop catalysts for conversion of fats, oils and fatty acid methyl esters into fungible diesel fuel

While triglycerides and fatty acid methyl esters (the primary components of vegetable oils and biodiesel, respectively) can be combusted alone or in mixtures without chemical modification, the physical properties of these compounds lead to problems related to cloud point, pour point, and high viscosity, and the chemical properties cause stability problems (typically susceptibility to oxidation). Task B studies involved synthesis, characterization and testing of heterogeneous catalysts for deoxygenation of both methyl esters and triglycerides. Both supported metal catalysts and zeolites were investigated. Effects of temperature, hydrogen pressure, and reactant chain length were investigated, and catalyst deactivation was measured.

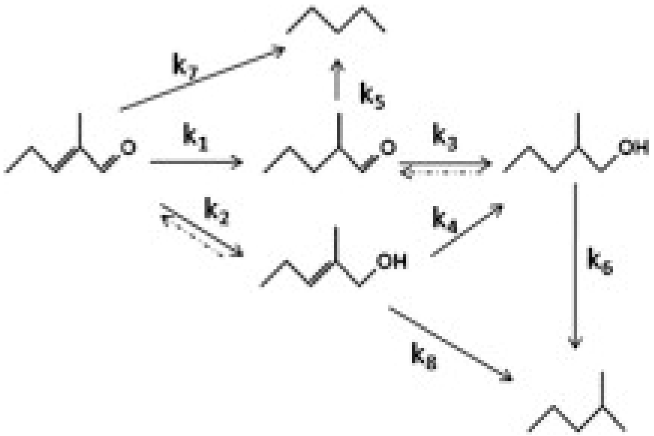
Over Pt/Al2O3 catalyst, in both vapor phase and liquid phase, product selectivity depended significantly on hydrogen partial pressure (Do, 2009). In the presence of He only, condensation and ketonization reactions took place resulting in oxygenated products, whereas selectivity to paraffins was high in the presence of H2. The results suggested that diesel products could be produced directly from the methyl esters. Over acidic zeolite catalysts, both straight chain and aromatic hydrocarbons were produced during conversion of methylesters; by comparison with reactions of the corresponding alkane, the reaction pathway leading to aromatic products was elucidated (Danuthai, 2009). The catalytic activity of the zeolite was also modified by exchange with Zn ions (Danuthai, 2009), which is known to improve aromatization activity with alkane feeds. The effect of Zn promotion was less with methylester feeds, however. Cs-exchanged X-zeolite was also studied as a potential catalyst for deoxygenation of methylesters to hydrocarbons suitable for diesel fuels in the absence of co-fed hydrogen (Danuthai, 2011). Over the CsNaX catalyst, methyl octanoate was observed to convert to heptenes and hexenes, probably by decomposition of an adsorbed octanoate-like species. Studies suggest that the Cs both increases the basicity of the catalyst and that extra framework Cs affects shape-selective features of the zeolite pores.

We also investigated direct catalytic conversion of triglycerides, which would eliminate the transesterification step (Chiappero, 2011). This research also investigated the possibility of production of higher-value products, in this case α-olefins, using reactive distillation to remove the primary conversion product α-olefin prior to double bond isomerization.

Task C: Fundamental catalytic studies to develop catalysts for conversion of glycerol and other small oxygenated compounds into fungible gasoline compounds

A significant fraction of pyrolysis oil products are oxygenated compounds (aldehydes, ketones, and carboxylic acids) with 1 to 5 carbon atoms. These molecules are smaller than gasoline or diesel requirements, so conversion processes other than hydrotreating or deoxygenation are required to utilize this fraction of the pyrolysis oil for fuel purposes. Very closely related is the conversion of glycerol, a 3-carbon oxygenate, to fungible fuels. Glycerol is the byproduct of biodiesel production via transesterification. Our studies under Task C investigated the use of solid acid and/or solid base catalysts as well as supported metal catalysts, and either physical mixtures of two catalysts or dual bed catalytic reactors. This task also included studies of process conditions to convert these molecules to suitable fuel compounds. Using these catalysts, larger carbon-number molecules can be created via condensation, ketonization, esterification or alkylation reactions; these molecules then may be treated along with other pyrolysis oil oxygenates as described in Task A.

Aldol condensation of glycerol (the major byproduct of biodiesel production from triglycerides) produces 2-methyl-2-pentanal, a six-carbon oxygenate. This aldehyde contains two reactive functional groups, i.e., both the C=O double bond and the C=C double bond, a characteristic that is also found in the oxygenate fractions of bio-oils produced from pyrolysis. During hydrotreating, it would be desirable to selectively remove only the oxygen without loss of carbon. Possible reaction pathways are shown below (Pham, 2009):

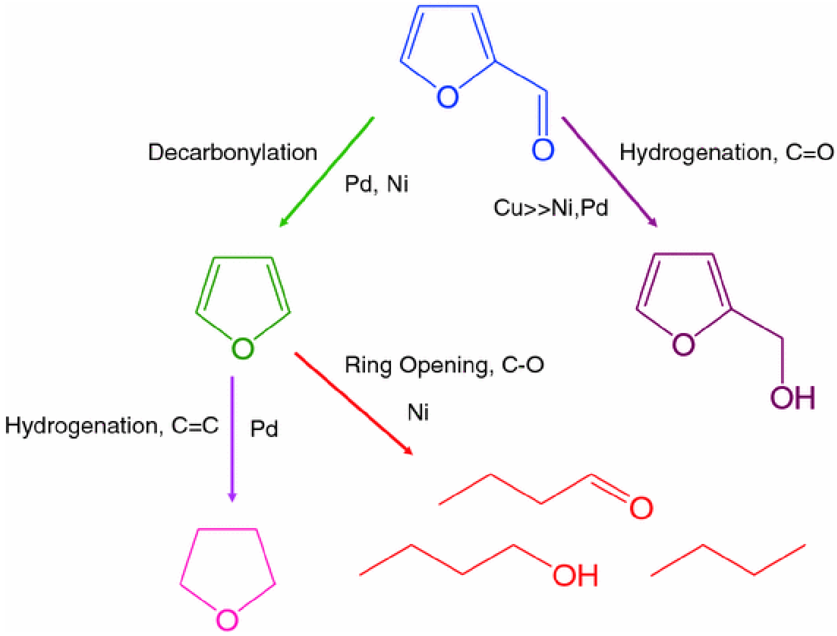


Hydrodeoxygenation and hydrogenolysis of 2-methyl-2-pentenal were studied on supported Pt, Pd, and Cu catalysts. The activity followed the order Pt > Pd > Cu. Major products were 2-methyl-pentanal, 2-methyl-2-pentenol and 2-methyl-pentanol. *n*-Pentane was produced on Pt and Pd while 2-methyl-pentane is produced on Cu. At very low conversion, Cu showed strong initial hydrogenation activity of C=O to form 2-methyl-2-pentenol. At higher temperatures over Cu, the hydrogenolysis of 2-methyl-pentanol took place, giving 2-methyl-pentane (and H_2O) as the dominant product while no decarbonylation is observed, thus preserving 100% of the carbon in the final hydrocarbon.

Conversion of C3 oxygenates was investigated over a number of catalysts and following a number of different chemical conversion steps. As already noted, glycerol (from biodiesel production) is such a C3 oxygenate, but a significant fraction of C3 carbon oxygenates is also produced during pyrolysis or torrefaction of lignocellulosic biomass. Moreover, large quantities of acetic acid are formed during pyrolysis or torrefaction, and acetic acid is readily ketonized to form acetone – another C3 oxygenate available for upgrading. HZSM-5 was found to effectively convert propanal (the primary glycerol dehydration product) to aromatic hydrocarbons. In fact, propanal was found to be much more reactive than propylene and to form mostly 2-methyl-2-pentenal and C_9 aromatics as early products in the reaction network through the aldol condensation pathway (Hoang, 2010). To better understand the ring-closure mechanism, the mesolite pore size and structure was varied by desilication (Zhu, 2010a), and to better understand and control deactivation, zeolite crystallite size was varied as well (Zhu, 2010b). Glycerol was directly converted to aromatic hydrocarbons over a series of zeolite catalysts (HZSM-5, HY, Mordenite, and HZSM-22) at 300−400 °C and atmospheric pressure or 2 Mpa (Hoang, 2010). The pore structure of the zeolite was found to play a significant role on the final product distribution. The major products over one-dimensional zeolites Mordenite and HZSM-22 are oxygenates (propenal, acetol, and heavy oxygenates) without aromatic formation. HZSM-22 is suitable for the production of acrolein with 86% yield at 100% glycerol conversion. However, it was found that glycerol can be converted to high yields of alkyl-aromatics, mainly C_8−C_{10} over three-dimensional HY and HZSM-5, with a maximum yield of 60% over HZSM-5. A two-bed configuration with a deoxygenation/hydrogenation catalyst (Pd/ZnO) as the first bed and HZSM-5 as the second bed can further increase the alkyl-aromatic yield. The mono-functional oxygenates produced by the first bed were oligomerized and aromatized into gasoline-range alkyl-aromatics over the second bed.

Another explored route to fungible fuel molecules is etherification. Dialkyl ethers were selectively produced from etherification of 2-methylpentanal with 2-methylpentanol at a molar ratio 1:1 at 125 °C (Pham, 2010). A yield of 79% ether with a selectivity of 90% was observed. Cross etherification of n-butanol with 2-methylpentanal shows a much higher rate than that observed when the alcohol or aldehyde is fed alone. This enhanced activity is in line with the catalyst requirement for large ensembles that allow surface alkoxide species next to η^2 adsorbed aldehydes. The dependence of the ether yield on metal loading suggests that etherification is highly sensitive to metal particle morphology, consistent with needing ensembles that accommodate the two adjacent adsorption sites.

Furfural and other furanic compounds are also found in significant concentration in pyrolysis oil. As with the smaller oxygenates, a significant challenge in upgrading of these compounds is maximizing carbon capture by selective conversion. Observed reaction pathways are shown in the figure below, from Sitthisa (2011a). In this and a subsequent paper (Sitthisa, 2011b), we reported that the distribution of products is a strong function of the metal catalyst used. High selectivity to furfuryl alcohol is obtained over Cu/SiO_2, with the formation of only small amounts of 2-methyl furan (the most desirable product which retains all carbon) at the highest reaction temperature studied. In contrast to Cu catalyst, the conversion of furfural over Pd/SiO_2 mainly produced furan by decarbonylation. Furan can further react with hydrogen to form tetrahydrofuran (THF). Finally, on Ni/SiO_2 catalysts ring opening products (butanal, butanol and butane) were obtained in significant amounts. The different product distributions were explained in terms of the strength of interaction of the furan ring with the metal surface and the type of surface intermediates that each metal is able to stabilize. Selective conversion of furfural to methylfuran was obtained over Ni-Fe bimetallic catalysts (Sitthisa, 2011b), with methylfuran yields approaching 40%. The addition of Fe suppresses the decarbonylation activity of Ni while promoting the C=O hydrogenation (at low temperatures) and the C-O hydrogenolysis (at high temperatures). DFT analysis of the possible surface species on the mono- and bimetallic surfaces suggests that the differences in selectivity displayed by these catalysts can be attributed to the stability of the η^2-(C,O) surface species, which is greater on the Ni-Fe than on pure Ni. As a result, this η^2-(C,O) species can be readily hydrogenated to furfuryl alcohol and subsequently hydrogenolyzed to 2-methylfuran on the bimetallic alloy due to a strong interaction between the carbonyl O and the oxyphilic Fe atoms.



A major scientific finding of Tasks A-C is the identification of catalysts and reaction conditions for high carbon-capture conversion of most of the oxygenates resulting from biomass pyrolysis or from conversion of vegetable oils. These conversion processes were demonstrated using model compounds, and in most cases reaction pathways and mechanisms were elucidated to facilitate understanding and improvement of the processes.

Based on these findings, novel improvements in biomass conversion and upgrading were proposed. Novel catalytic systems such as Janus catalyst particles in biphasic reaction systems were explored for their potential for improved carbon capture, catalyst stability, and combined reaction/separation characteristics to enable process intensification (Shen, 2009;Crossley, 2010; Faria, 2010; Pilar Ruiz, 2011; Fan, 2011; Zapata, 2012). Our research findings also led to the exploration of staged torrefaction/pyrolysis as a thermal degradation process to develop simpler intermediate streams to facilitate upgrading with catalytic processes designed to maximize carbon capture and minimize hydrogen consumption. These studies became a major focus in the DoE Carbon, Hydrogen and Separations (CHASE) project subsequent to this project.

Task D: Measurement and Prediction of Biofuel Combustion Characteristics

Knowledge of the combustion and pollutant emission characteristics is an important part in the development and application of biofuels. Previous engine studies have indicated that the use of biodiesels, for example, is accompanied with an increase in NO_x emission, but the observed NOx results have been controversial and sometimes opposite to each other. Hence, these results cannot be used reliably to predict the increase/decrease in NO_x formation with changes in the properties of fuels. In order to gain a fundamental understanding of the combustion behavior of biofuels, detailed studies were carried out under controlled conditions, followed by engine studies.

The degree of unsaturation of methyl esters is known to affect the pollutant emissions. We studied laminar partially premixed flames to investigate the effect of iodine number on NO_x formation using three vaporized biofuels: canola methyl ester, soy methyl ester, and methyl stearate (Love, 2009; Love, 2011). The iodine numbers for the selected fuels varied over a wide range from 0.5 to 141. Key measurements included NO_x concentration and temperature fields. We observed that the peak NO_x concentration significantly increased with the iodine number, indicating a strong correlation between the chemical structure of the fuel and NO_x emission. Additional studies were carried out in a small-scale gas turbine (Habib, 2010). The addition of biofuel resulted in a reduction in static thrust and thrust-specific fuel consumption, and increased thermal efficiency. The CO and NO emissions from the turbine were reduced with the biofuel blends. The results suggest that an optimum mixture may be found that reduces pollutant emissions while producing the desired thrust. Further studies of biofuel properties showed that CME biofuel spray flames produced lower in-flame NO and CO peak concentrations than No. 2 diesel fuel spray flames (Erazo, 2010a) and that, at identical volumetric flow rates of fuel, atomization air and co-flow air, a canola methyl ester biofuel produced lower in-flame NO and CO peak concentrations than No. 2 diesel fuel (Erazo, 2010b).

Characteristics of biofuels in a diesel engine at partial load were also studied (Sequera, 2011), particularly to determine if the higher emissions of unsaturated fuels can be compensated for by engine operating characteristics, specifically fuel injection timing. We found that late injection timing of the engine provided advantages in the CO and NO emissions with a small penalty in fuel consumption and thermal efficiency.

Other studies characterized combustion characteristics of biofuels in porous media burners (Barajas, 2012) and in turbulent gas jet flames (Parthasarathy, 2013), and investigated important fire safety characteristics of biofuels and their blends with petroleum diesel (Tran, 2014).

Task E: Development of quantitative structure-property relationships relating fuel properties to chemical structure

The initial intent in this task was to analyze the relationships between the molecular structure and specific chemical properties relevant to fuel characteristics (octane number, cetane number, combustion efficiency, soot and emissions characteristics). For our research program, however, we found immediate need for similar relationships related to gas chromatographic analysis. Fastpyrolysis oil has a complex composition that includes acids, aldehydes, furans, sugars, alcohols, ketones and phenolic compounds that are very reactive and difficult to analyze by chromatographic techniques. By building databases that include the Relative Retention Time (RRT) and Relative Response Factor (RRF) from oxygenated compounds, Quantitative Structure Property Relationships (QSPR) were developed to identify and quantify compounds present in the pyrolysis bio oil as well as intermediate products obtained during the bio oil conversion to fuels. We found the greatest utility by developing separate relationships for each of two particular compound groups: aliphatic oxygenates (AO) and Phenolic and Heterocyclic Oxygenates (PHO), as illustrated below.

Data of relative response factors (RRF) and relative retention times (RRT) for aliphatic oxygenates are presented in the table below:

Molecules	RRT	RRF
2-butanone	1.20	1.94
1,5-pentanediol	5.23	3.70
1-dodecanol	5.05	4.57
glycerol	5.65	0.96
1-propanol	1.62	2.08
1,2-propanediol	4.48	1.75
3-pentanol	1.88	2.40
cis-2-buten-1,4-diol	5.23	1.76
Acetone	1.00	1.41
6-undecanone	3.66	4.13
cyclohexanone	2.72	2.83
cyclopentanone	2.24	2.46
2-methyl-3-pentanone	1.50	2.33
2,5-hexanedione	3.59	2.13
2-hexanone	1.81	2.48
4-heptanone	1.98	3.11
3-pentanone	1.42	1.24
2-methyl-1-pentanol	2.68	1.68
2-octanol	3.20	1.75
1,4-butanediol	4.84	1.23

Experimental RRT and RRF values for selected aliphatic oxygenates

Using data showed in the table, and molecular descriptors calculated by QSAR MDL, Quantitative Structure-Property Relationships were developed in order to predict RRT and RRF of oxygenated aliphatic compounds consisting of ketones and alcohols, since alcohols constitutes 2-5% and ketones 1-5% of bio composition; additionally, some of derivative species formed during bio oil upgrading have ketone and alcohol functionalities..

QSPR for RRT was done using as descriptors: molecular weight (fw), molecular and group polarity index (Qv) and number of hydrogen bond donors (numHBd). The model has the following form:

$$RRT_{AO} = -2.016*Qv + 0.03346*fw + 1.044*numHBd + 1.43825$$

Accuracy of the aliphatic oxygenates RRT model was tested by calculating statistical parameters such us P-value (P), coefficient of determination (R^2), crossed squared correlation coefficient (Q^2) and standard deviation (S). Results are shown below:

R^2	0.978
S (error)	0.268
P	3.565E-10
Q^2	0.948

Statistical parameters of the RRT model of aliphatic oxygenates.

The model has a high R^2 and Q^2 while P and S are small, indicating that the QSPR proposed has good predictive ability. The figure below graphs the measured versus predicted RRT values.

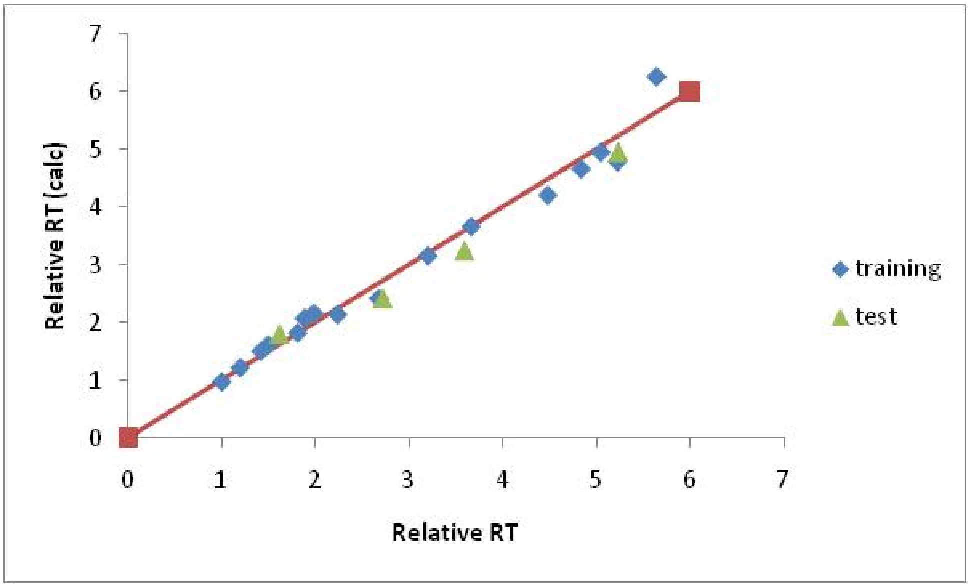


Figure E.1. Comparison of experimental and predicted data for RRT of aliphatic oxygenates.

Using the experimental measurements of GC residence time, a QSPR to predict RRF of aliphatic oxygenates was proposed taking into account the molecular descriptors: sum of delta intrinsic states of atoms in the molecule (sumdelI), valence zero order chi index (x0), valence 1st order chi index (xv1) and molecular and group polarity index (Qs).

$$\text{Relative } RF_{AO} = -0.8439*sumdelI - 3.435*Qs + 0.747*x0 + 3.324*xv1 - 0.640125$$

Statistical measures of the “goodness of fit” of this model are presented below; as with the case of residence time, the relative response factor is well-predicted.

R^2	0.987
S (error)	0.142
P-value	1.31E-7
Q^2	0.959

Statistical parameters of the Relative Response Factor (RRF) model of aliphatic oxygenates.

The phenolic and oxygen heterocycle compounds constitute as much as 9% of the bio oil composition, with phenolics 2-5 % and oxygen heterocycle 1-4% [**Error! Bookmark not defined.**]. Experimental data of RRT and RRF for this group of compounds was also obtained analogous with what was done for the aliphatic oxygenates. Using this data, a quantitative structure property relationship was obtained. Descriptors selected for RRT QSPR of phenolic oxygen-heterocycle compounds were the valence zero order chi index (x0), molecular weight (fw) and the largest positive charge over the atoms in molecule (MaxQp). Model results are summarized following:

$$RRT_{POH} = 1.735*x0 - 0.05773*fw + 20.75*MaxQp - 3.22413$$

R^2	0.949
S (error)	0.491
P-value	9.208E-7
Q^2	0.906

Statistical parameters of Relative Residence Time (RRT) model for phenolic and heterocyclic compounds.

For the response factor, the descriptors that offered a better correlation were largest positive charge over the atoms in molecule (MaxQp) and number of hydrogen bond acceptors (numHBa). The QSPR model for RRF was:

$$RRF_{POH} = 5.773*MaxQp - 1.065*numHBa + 2.65902$$

The table below indicates this model is not as good as the one presented for the aliphatic oxygenates, since the coefficient of determination (R^2) is lower and the standard deviation (S) is bigger than in the aliphatic oxygenates model, possibly due to the complexity of the interactions of phenolic heterocycle with the column coating.

R^2	0.875
S (error)	0.473

P-value	7.31E-4

Statistical parameters of RRF model of phenolic oxygen heterocycle compounds.

QSPR constitutes a valuable tool to predict properties of compounds. In the case of bio oil analysis it is very important since it can eliminate much of the guesswork currently associated with GCMS spectroscopy and the bio oil mixtures can be more readily and accurately analyzed.

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“Catalytic Conversion of Lignin Fractions of Biomass,” Daniel E. Resasco, Lance Lobban, Richard Mallinson, Xinli Zhu,Teerawit Prasomsri, Lei Nie and Sunya Boonyasuwat, AICHE Annual Meeting, Salt Lake City, UT, November 2010.

“Understanding Deoxygenation Reactions to Produce Fungible Transportation Fuels On An Optimized Supported Pt Catalyst,” Kyle W. Elam, Phuong T. Do, Min Shen, Rolf Jentoft, L.L. Lobban and Daniel E. Resasco, AICHE Annual Meeting, Salt Lake City, UT, November 2010.

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