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Solvatochromic rationalization of co-solvent effects on the synthesis of biodiesel

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Abstract

Biodiesel is a promising fuel due to its environmental benign nature. However, there are lots of challenges inherent in the quantum and quality of synthesized biodiesel due to the influence of multi-reaction conditions and co-solvents effects on biodiesel synthesis. This work however investigates the effect of selected solvents on solvent solvatochromic properties. Empirical models were developed to establish quantitative relationships between the solvatochromic properties on biodiesel yield both for homogeneous and heterogeneous catalysis. For the later, IBM SPSS was used to perform regression analysis, yielding a regression model with an R^2 value of 0.985. The results indicate that biodiesel yield increases with higher solvent dipolarity/polarizability (π^*), acidity (α), and dielectric constant (ϵr), while it decreases with higher normalization value (ETN), refractive index (n), and basicity (β). For the homogeneous catalyst, machine learning was employed using Google Colab Jupyter Notebook, resulting in a regression model with an R^2 of 1.00, indicating perfect predictive accuracy. In contrast to the heterogeneous catalyst model, ETN, β, and ϵr had the most significant positive effects on biodiesel yield, whereas acidity (α), dipolarity (π^*), and refractive index (n) negatively impacted biodiesel yield.The findings highlight distinct solvent effects depending on the catalyst type and provide a systematic framework for solvent selection in biodiesel production. The developed models can be applied to predict biodiesel yield for various solvents, enabling the selection of optimal co-solvents for enhanced biodiesel efficiency and sustainability.

Nomenclature and units

α	Acidity
β	Basicity
π^*	Dipolarity
n	Refractive Index

1.0 Introduction

Fuels are substances that stored energy in chemical or nuclear forms which can be released to perform work through combustion, chemical reactions, nuclear or other processes (Kumar, 2016, kumar et al.,2020).One of such fuels is the fossil fuel and as energy carriers, such fuels facilitate the conversion of energy from one form to another. However, the availability of fossil fuels is expected to decrease in the near future due to the depletion of oil reserves.(Rashid et al., 2017). In addition, the excessive production and consumption of fossil fuels have caused numerous global issues, to include climate change, environmental degradation, adverse effects on public health, increased greenhouse gas emissions among others. (Oa et al., 2019).To address these challenges, several alternatives to fossil fuels have emerged to meet global energy demands, while mitigating their environmental impacts. These alternatives include renewable energy sources such as wind, solar, hydroelectric, and geothermal power, as well as biofuels like ethanol and biodiesel(Luu et al., 2014a). Other viable options include green hydrogen produced through the electrolysis of water, and nuclear energy. Among biofuels, biodiesel, derived from organic waste as plants, animals, algae, and industrial wastes, has proven to be highly promising(Singh et al., 2022). Biodiesel, bioethanol, biohydrogen, and biogas are other types of biofuels available. This study focuses on biodiesel as an alternative energy source to fossil fuels. The American Society for Testing and Materials (ASTM) defines biodiesel as a blend of non-alkyl esters of long-chain fatty acids, generally sourced from oils from plants or animal fats.(Ali Mohammed-Dabo et al., 2012). These sourced oils can be non-edible, such as castor and jatropha oils, or edible, like soybean oil. Currently, over 95% of biodiesel production globally relies on edible oils. Biodiesel has gained significant attention worldwide due to its favorable properties, including renewability, biodegradability, non-toxicity, and excellent lubricity. It is also nearly free of sulphur and aromatic compounds, making it an environmentally friendly alternative suitable for use in diesel engines (Thirumarimurugan et al., 2012)

The production of biodiesel primarily involves transesterification process. This method faces challenges such as selecting suitable feedstocks and catalysts, achieving efficient production methods, and managing high production costs.(Abdulkadir et al., 2018). Feedstock selection, in particular, greatly impacts the cost of biodiesel. Other issues include slow reaction rates, incomplete conversion of triglycerides, poor separation, soap formation, and high energy consumption.(Díaz et al., 2023) Exploring innovative solutions, such as using co-solvents, can help overcome these challenges. Co-solvents act a massive role in improving reaction efficiency, especially, when reacting feedstocks with high free fatty acid content or less reactive alcohols. By improving the solubility and the interaction between oil and alcohol, co-solvents speed up the rate of reaction (Pasham et al., 2020). Some co-solvents include, tetrahydrofuran (THF), acetone, and diethyl ether, these co-solvents create a homogeneous phase, enhance mass transfer, and reduce reaction time(Thanh et al., 2013). This lower energy requirement resulted in higher yield of biodiesel produced, Also, co-solvents accelerate phase separation between fatty acid methyl esters (FAME) and by-products after the reaction is complete (Viraj Miyuranga et al., 2022).

The effect of solvents on biodiesel synthesis rates was first observed by Berthelot and Pean de Saint-Ailles in 1862, when, they were working on the esterification of acetic acid with ethanol. The solvatochromic comparison method provides a model for examining and measure solvent effects through solvatochromic parameters such as, acidity (α), dipolarity (π*), and basicity (β) (Rizka Amaliah et al., 2020). These parameters evaluate solvent's ability to strengthen charges, donate or accept protons, and facilitate interactions within the reaction medium. For example, dipolarity is proportional to a solvent's molecular dipole moment, while the acidity and basicity scales reflect its hydrogen-bonding capabilities (Kamlet et al., 1983a). Additional solvatochromic parameters, including electrophilicity (ET30), normalized electronic transition energies (ETN), dielectric constant, and refractive index, further characterize solvent behavior (Kamlet et al., 1983a).

Rationalizing the use of co-solvents entails selecting solvents that optimize reaction conditions by enhancing the solubility of reactants, improving mass transfer, and minimizing reaction times and energy consumption. The objective herein is to identify co-solvents that strike a balance between cost, safety, environmental impact, and performance. A thoughtful selection of co-solvents can greatly enhance the transesterification process, resulting in more efficient and sustainable biodiesel production (El Seoud et al., 2024).

2.0 Materials and Methods

International Business Machines Statistical Package for Social Sciences (IBM SPSS) and Python codes were employed to model a linear regression equation to determine the optimal framework for selection of co-solvent for biodiesel synthesis.

3.0 Data Collection

Data collection involves selecting and characterizing a diverse set of solvents, each defined by specific solvatochromic properties. Literature sources detailing the use of these co-solvents in biodiesel synthesis were sourced and utilized for this work.

i. Biodiesel Yield Data

The biodiesel yield data obtained from the synthesis using selected co-solvents, were sourced from the works of (Díaz et al., 2023) and (Encinar et al., 2016). The papers provide experimental results on biodiesel yield from various feedstocks, catalysts, co-solvents, and reaction conditions.

ii. Solvatrochromic Parameters of Co-solvent

The solvatochromic parameters of the co-solvents used in this study were sourced from literature (Kamlet et al. 1982 and Reichardt,1994) The selected solvents include acetone, hexane, diethyl ether (DEE), tetrahydrofuran (THF), dimethyl ether (DME), diisopropyl ether (DIBE), diisopropylether (DIIPE), methyl tert-butyl ether (MTBE), toluene, tert-butyl methyl ether (TMBE), 1-propanol, and 2-propanol.

Tables 1 and 2 present the co-solvents, their corresponding solvatochromic parameters, and the corresponding biodiesel yield obtained for both homogeneous and heterogeneous catalysts respectively

Table 1: Parameters of Co-solvents and Biodiesel Yield for Heterogeneous conditions

Co-solvent	π*	α	B	ϵr	N	ET(30)	ET^N	Yield
Acetone	0.71	0.08	0.48	20.70	1.36	42.20	0.355	85.30
THF	0.58	0.00	0.55	7.58	1.41	37.40	0.207	86.00
Hexane	-0.08	0.00	0.00	1.89	1.37	31.00	0.009	97.03
2-prop	0.48	0.76	0.95	18.30	1.38	48.40	0.550	98.30
1-prop	0.52	0.78	0.00	20.10	1.39	50.70	0.620	95.69
Toluene	0.54	0.00	0.11	2.38	1.50	33.91	0.100	90.30
DEE	0.27	0.00	0.47	4.33	1.36	34.50	0.1170	87.60
TMBE	0.37	0.06	0.00	7.60	1.36	34.70	0.124	97.50

Table 2: Parameters of Co-solvents and Biodiesel Yield for Homogeneous conditions

Co-solvent	π*	α	B	ϵr	n	ET(30)	ET^N	Yield
MTBE	0.30	0.00	0.41	4.60	1.369	42.30	0.284	97.70
DIIPE	0.27	0.00	0.49	4.30	1.404	34.10	0.105	87.30
ACETONE	0.71	0.08	0.55	20.70	1.360	42.20	0.350	97.91
HEXANE	-0.08	0.00	0.00	1.89	1.370	31.00	0.009	94.14
THF	0.58	0.00	0.55	7.58	1.410	37.40	0.207	96.00
DEE	0.27	0.00	0.47	4.33	1.360	34.50	0.117	98.35
DME	0.12	0.18	0.20	6.30	1.310	32.40	0.221	99.00
DIBE	0.24	0.00	0.41	4.20	1.411	33.00	0.071	74.30

4.0 Linear Regression using IBM SPSS

IBM SPSS version 27.0.1 was installed to perform statistical analysis for this study. Upon installation, the software was launched, and a spreadsheet workspace appeared, into which all data were manually entered. The data was input by directly typing values into the respective cells of the spreadsheet. Linear regression analysis was performed using SPSS's statistical features of IBM SPSS. The process began by navigating to the 'Analyze' menu, selecting 'Regression', and then choosing 'Linear'. In the Linear Regression dialog box, the dependent variable, biodiesel yield, was specified, while the solvatochromic parameters were designated as independent variables. Additional output options, such as confidence intervals and model fit statistics, were selected before running the analysis. The SPSS generated output tables that included the regression coefficients, significance values (p-values), R-squared, adjusted R-squared, and ANOVA results. These outputs were used to evaluate the strength and validity of the relationship between the dependent and independent variables, providing valuable insights into the influence of solvatochromic parameters on biodiesel yield.

5.0 Development of Empirical Model

Based on the regression coefficients obtained from the linear regression analysis, an empirical model of the form of Eq.1 was developed to predict the biodiesel yield as a function of the solvatochromic parameters of the co-solvents. The general form of the equation is expressed as:

$$\text{Yield} = \beta 0 + \beta 1 \cdot \text{Parameter1} + \beta 2 \cdot \text{Parameter2} + \cdots + \beta n \cdot \text{Parameter n} \quad \text{----------(1)}$$

β0 represents the intercept, which is the predicted biodiesel yield when all the independent parameters are equal to zero.

β1,β2,…,βn are the regression coefficients corresponding to each solvatochromic parameter(Parameter1,Parameter2). These coefficients indicate the strength and direction of the relationship between each parameter and the biodiesel yield.

6.0 Model Validation

The ability of the empirical model to predict biodiesel yield for solvents not initially included in the dataset is a critical test of its robustness and generalizability. When the solvatochromic parameters of an additional solvent were provided, the derived model can be used to predict the biodiesel yield for that solvent. This process is essential for evaluating the strength of the model particularly its performance beyond the initial dataset and whether it can be reliably applied to new scenarios.

7.0 Data Analysis using Machine Learning

For a broader modelling view in line with the current global and best practices, Google Collab was further utilized as the primary platform for data analysis, with the study focusing

on the application of linear regression. The analysis commenced with data pre-processing, which involved cleaning and organizing the dataset to ensure its completeness and accuracy. Python libraries, including pandas and numpy, were employed for efficient data manipulation and handling. Exploratory data analysis was conducted using matplotlib and seaborn, enabling the identification of trends, patterns, and relationships within the data. The linear regression model was developed (Eq.2) in terms of solvatochromic parameters such as **π*** (dipolarity/polarizability), **α (acidity), β (basicity),εr** (dielectric constant),**n** (refractive index),**ETN** (normalization value) and implemented using the scikit-learn library. This facilitated the prediction of the dependent variable based on the independent variables. Model evaluation metrics, such as R-squared and Mean Squared Error, were applied to assess the predictive accuracy and reliability of the model. The coefficients of the regression model were subsequently analyzed to extract meaningful insights and support data-driven conclusions.

$Yield$ = 177.742+ 11.425π∗+ 51.307α – 2.357β +0.325ϵr – 44.904n – 0.590E(30) −68.444ETN (Eq.2)

8.0 Results and Discussions

The coefficient of determination (R^2=0.985) indicates that this equation explains 98.5% of the variation in biodiesel yield, signifying a high level of predictive accuracy. The equation models biodiesel yield as a function of solvent parameters, with the results showing that the solvent's **α (acidity)** parameter has the largest positive effect on yield, followed by **π*** (dipolarity/polarizability) and **εr** (dielectric constant). In contrast, **ETN** (normalization value) exerts the largest negative influence on biodiesel yield, followed by **n** (refractive index), and **β (basicity)**. A unit increase in **ETN**, **n**, and **β** leads to a reduction in biodiesel yield by 68.44, 44.904, and 2.357 units, respectively. On the other hand, a unit increase in **π***, **α**, and **εr** will increase the biodiesel yield by 11.425, 51.307, and 0.590 units, respectively. These findings suggest that biodiesel yield increases when solvents exhibit higher **dipolarity/polarizability, dielectric constant**, and **acidity**, but lowe**r normalization** values and refractive index.

When comparing these results with those of (Owolabi et al., 2016a) , who modeled the effect of solvents on styrene monomer conversion in polymerization, some similarities and differences emerged. (Owolabi et al., 2016a) used IBM SPSS to model styrene conversion and obtained a regression coefficient of (R^2=0.945), which explains 94% of the variation in conversion. Their predictive equation is shown in Eq.3

X = 4.407 +4.265$\pi*$ −1.843α −3.354β −0.018E −4.365n +4.205ETN, ----------(Eq.3)

Owolabi et al.,2015 reported that, the **π*** (dipolarity/polarizability) parameter has the largest positive effect on styrene conversion, followed by **ETN**, while the **refractive index (n)** showed the largest negative influence, followed by **basicity (β)** and **acidity (α)**. A unit increase in **α**, **β**, **Er**, and **n** leads to reductions in styrene conversion by 1.843, 3.354, 0.018, and 4.365, respectively. Conversely, a unit increase in **π*** and **ETN** increases conversion by 4.265 and 4.205, respectively. Owolabi's results suggest that styrene conversion increases with solvents that exhibit high **dipolarity/polarizability** and **ETN**, but with a low **refractive index**.

Comparing the two sets of results, it is evident that both studies highlight the significant role of **dipolarity/polarizability (π*)** and **normalization value (ETN)** in influencing solvent performance, albeit with some differences in the effect of **acidity (α)** and **refractive index (n)**. For both biodiesel yield and styrene conversion, solvents with higher **dipolarity/polarizability** and **ETN** lead to better outcomes, while solvents with lower **refractive index** tend to perform better. The primary difference lies in the influence of **acidity (α)**, which has a more pronounced positive effect on biodiesel yield in this study, while it exerts a negative effect on styrene conversion in Owolabi et al.'s 2016 study. This suggests that the specific application (biodiesel production vs. styrene polymerization) may influence the way solvent properties impact the reaction, reflecting different solvent-solute interactions in these processes.

For homogeneous catalyst and solvatchromic parameters of selected solvents, both machine learning and IBM SPSS were used, with the two, we arrived at the same model (Eq.4).

The final equation;

$Yield$ = 1259.7584 – 198.5388π∗ – 1547.2566α + 9.5034β +6.2074ϵr – 126.2578n – 33.3676E(30) + 1570.4607ET^N ----------(Eq.4)

The regression analysis was implemented using a machine learning model with a 95% confidence level, achieving a coefficient of determination (R^2= 1.00), This indicates that the model explains 100% of the variation in biodiesel yield based on the solvent parameters.

From the regression equation, it is evident that ETN has the most significant positive effect on biodiesel yield, followed by β (basicity) and εr (dielectric constant). Conversely, the solvent parameter α (acidity) exerts the largest negative

influence, followed by π∗ (dipolarity/polarizability), refractive index (n), and electrophilic parameter ET(30).

Quantitatively, a unit increase in π∗, α, n, and ET(30) reduces biodiesel yield by factors of 198.5388, 1547.2566, 126.2578, and 33.3676, respectively. On the other hand, a unit increase in ϵr, β, and ETN enhances the biodiesel yield by factors of 6.2074, 9.5034, and 1570.4607, respectively. These findings indicate that biodiesel yield increases with solvents characterized by higher ϵr, β, and ETN.

Bini et al.,2008 analyzed solvent effects on the Diels-Alder reaction in ionic liquids, emphasizing that electrophilicity (ETN) and polarity (π∗) significantly impacted reaction rates and selectivity. This study, ETN also emerged as a crucial positive factor for biodiesel yield, consistent with Bini et al.'s findings on its role in enhancing reactivity. However, while Bini et al. observed that high π∗ values favored certain reactions, this study results suggested that π∗ negatively affects biodiesel yield, possibly due to differences in reaction mechanisms or other effects between the two processes.

The linear regression equations correlation established in this study can be used to predict the yield achievable from any solvent, provided the KT parameter of such solvent are known or can be determined experimentally, and thus enable evaluation of its suitability.

The regression model explains 98.5% of the variance in biodiesel yield (R^2 = 0.985), indicating a strong fit. The adjusted R^2 (0.787) accounts for the sample size and predictors, showing good accuracy. The low standard error (2.53901) suggests reliable predictions, and the significant F Change (5.305) confirms that the predictors meaningfully contribute to explaining yield. This model performs well, but it would benefit from a larger sample size for more robust validation.

Table 3.0: Model Summary[b]

Model	Change Statistics	
	df2	Sig. F Change
1	1	.321

The **Change Statistics** table shows the results of the F-test for the model's overall significance:

df2 (1): This indicates that one predictor variable was added to the model.

Sig. F Change (0.321): This p-value is above the typical significance threshold of 0.05, suggesting that the addition of the predictor does not significantly improve the model. In other words, the new variable might not have a substantial impact on explaining the variation in yield. This indicates that, while the model fits well overall, the specific addition of this predictor might not contribute significantly.

9.0 Comparison of Solvatochromic Effects on Biodiesel Yield for Homogeneous and Heterogeneous Catalysis,

Solvatochromic parameters describe how solvents influence reaction outcomes based on their polarity, acidity, basicity, and other electronic properties. In this study, the effects of these parameters on biodiesel yield were analyzed for both **homogeneous** and **heterogeneous** catalysis, leading to distinct trends in their impact. Table 4 explains the comparison for both homogenous and heterogenous catalyst.

Table 4: Solvatochromic Effects on Biodiesel Yield for Homogeneous and Heterogeneous System.

Solvatochromic parameter	**Homogenous catalyst (effect on yield)**	**Heterogenous catalyst (effect on yield)**	**Summary of Findings**
ETN(Electophilicity)	+1570.4607 Strong positive	-68.444 Negative	Solvents with high ETN promotes heterogeneous system and hinders heterogenous system
π∗(Dipolarity or polarizability)	-198.5388 (Negative)	+11.425	Solvents with high π∗ improves heterogenous system but hinders homogenous catalysis
α (acidity)	-1547.2566 (strong negative)	+51.307 (positive)	Acidity negatively affects homogeneous catalysis but enhances heterogenous catalysis
β (Basicity)	+9.5034 Positive	-2.357 (Negative)	Basicity promotes homogeneous systems but hinders heterogenous systems
ϵr (Di electric constant)	+6.2074 Positive	+0.325 positive	Di electric constant improves biodiesel yield in both cases, but its effect is stronger in homogenous catslysis

n(refractive index)	-126.2578 Negative	-44.904 Negative	Refractive index negatively affects biodiesel yield in biofuel systems, but more significantly in homogeneous catalysis
ET(30) Electroplillic	-33.3676 Negative	-0.590 Negative	Electrophilic solvents reduce biodiesel yield in both cases, but their impact is stronger in homogenous catalysis

10.0 Conclusion

This study successfully developed regression models to predict biodiesel yield based on solvatochromic parameters for both homogeneous and heterogeneous systems. The homogeneous catalyst model, derived using machine learning, achieved a perfect predictive accuracy ($R^2 = 1.00$), indicating that 100% of biodiesel yield variation was explained by solvent properties. The most influential positive parameters for biodiesel yield in homogeneous catalysis were electrophilicity (ETN), basicity (β), and dielectric constant (εr), while acidity (α), dipolarity/polarizability (π*), refractive index (n), and electrophilic parameter ET(30) had negative effects.

In contrast, the heterogeneous catalyst model, derived using IBM SPSS exhibited a slightly lower predictive accuracy ($R^2 = 0.985$), demonstrating that 98.5% of biodiesel yield variation could be explained by the regression equation. The solvent parameters that positively influenced yield were acidity (α), dipolarity/polarizability (π*), and dielectric constant (εr), while normalization value (ETN), refractive index (n), and basicity (β) exerted negative effects. These results indicate that solvent effects on biodiesel yield depend significantly on the catalytic system used. While homogeneous catalysis benefits from solvents with high ETN and β values, heterogeneous catalysis requires higher acidity and dipolarity/polarizability for optimal yield. The empirical models serve as simple industrial tools free of usage ambiguities to pre-determine ideal co-solvents for biodiesel synthesis.

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Declaration of conflict of interest

The authors have collectively contributed to the conceptualization, design, and execution of this journal. They have worked on drafting and critically revising the article to include significant intellectual content. This manuscript has not been previously submitted or reviewed by any other journal or publishing platform. Additionally, the authors do not have any affiliation with any organization that has a direct or indirect financial stake in the subject matter discussed in this manuscript.

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