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Andri Cahyo Kumoro, Hadiyanto, Hong-Wei Yen,
Abdul Latif Ahmad, Oki Muraza, Xosè Antòn Vàzquez Àlvarez,
Ivan Salmeron Ochoa (Mexico)

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Preface

Preface

International Conference on Chemical and Material Engineering (ICCME) 2018

The 3rd International Conference on Chemical and Material Engineering (ICCME) 2018 was held by the Department of Chemical Engineering - Universitas Diponegoro in conjunction with the 60th anniversary of Faculty of Engineering - Universitas Diponegoro. The event was conducted on September 19th 2018 in Grand Candi Hotel Semarang - Indonesia. This event was the continuation of the preceding conferences held in 2013 and 2015. The ICCME 2018 brought "Chemical and Material Engineering for Sustainable Foods, Energy, and Environment" as the grand theme. The objectives of this conference are:

- To disseminate and develop the most recent innovations, trends, and concerns as well as practical challenges encountered and solutions in the fields of chemical and material engineering.
- To encourage international collaborations and joint ventures
- To promote and facilitate the growth of scientific and technical development in the field of chemical and material engineering development in Indonesia and the Asia region

Although the organizing committee received a total of 115 abstract papers, the scientific committee decided that only 97 papers were eligible for oral presentations which came from Indonesia, Malaysia, Myanmar, the Philippines, Taiwan, Japan and Austria. In addition, 3 keynote and 3 invited speakers from abroad were presented during this conference. This special issue proceeding presents 76 papers selected from the presented papers after being thoroughly peer reviewed.

Special thanks are due to all keynote and invited speakers for their participation, to the Organizing Committee members for all their hard work in making the conference to happen, especially Universitas Diponegoro Chemical Engineering Master Program students who generously supported the conference. The Scientific Committee members are also thanked for reviewing the submitted manuscripts and for assisting in the editorial process. Finally, all who travelled to Semarang, Indonesia for the meeting are acknowledged for deciding to attend and contribute to making this a successful conference.

We greatly hope that this proceeding will be advantageous in future implementations of chemical and material engineering, not only to the academia, but also to the industries by which broadening our scientific perspective.

Andri Cahyo Kumoro & Dyah Hesti Wardhani

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Simplex Lattice Design Method for the Optimization of Non-Edible Oils Mixture Composition as Feedstock for Biodiesel Synthesis Using Reactive Distillation

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Simplex Lattice Design Method for the Optimization of Non-Edible Oils Mixture Composition as Feedstock for Biodiesel Synthesis Using Reactive Distillation

R D Kusumaningtyas[1,*], B Triwibowo[1], R A Ramadhani[1], D H S Riyadi[1], I Istadi[2]

[1]Chemical Engineering Department, Faculty of Engineering, Universitas Negeri Semarang.
[2]Chemical Engineering Department, Faculty of Engineering, Diponegoro University, Semarang, Indonesia

Email: ratnadewi.kusumaningtyas@mail.unnes.ac.id

Abstract. The diminished of fossil fuel becomes a critical issue today. Thus, it is essential to develop alternative energy resources which are renewable and environmental friendly. Among the prospective alternative is biodiesel. Biodiesel (fatty acid methyl ester) is produced by combining vegetable oils with alcohol via transestefication reaction. To avoid conflict between food and energy needs, the uses of non-edible oils as raw material is suggested. Commonly, biodiesel synthesis employs a single type of vegetable oil feed-stock. The dependency on a certain feed-stock could cause an increasing price due to the high demand and low supply. Thus, it is beneficial to apply diversification by employing multiple feed-stocks or mixture of non-edible oils as biodiesel feedstocks. However, the best composition of the multiple feed-stocks is needed to obtain the optimum yield of biodiesel. In this work, mixture of non-edible oils, i.e. jatropha, nyamplung (*Calophyllum inophyllum L.*) seed, and used cooking oils were applied as biodiesel feedstocks. Non-edible oils are cheap but have high free fatty acid (FFA). The high FFA is not favorable for the alkaline-catalyzed transesterification since it will cause undesired saponification reaction and lower biodiesel yield. Hence, pretreatment to reduce FFA content is crucial prior to transesterification. In this work, esterification of FFA in vegetable oils mixture (jatropha, nyamplung, and used cooking oils) with methanol using reactive distillation in the presence of $SnCl_2$ catalyst was conducted as pretreatment step of biodiesel production. Various compositions of the oils mixture were employed. To determine the best formulation of feed-stocks, Simplex Lattice Design Method (SLDM) was used for the optimization of non-edible oils mixture composition as feedstock for biodiesel synthesis using reactive distillation. The prediction using SLDM was validated with the experimental data. It was found that the optimal composition of oil mixture was the mixture contains 37.291 v/v% jatropha oil + 37.709 v/v% nyamplung oil + 25 v/v% used cooking oil. This composition of feedstocks revealed the FFA conversion of 85.93% (prediction) and 86.03% (experiment). The desirability value was 0.937 which indicated the validity of the SLDM optimization method. The esterified oil mixture was then underwent transesterification reaction to produce biodiesel. The yield of biodiesel demonstrated 99.65 % purity and density of 884 kg/m^3.

1. Introduction

leading to the world's temperature. This condition will cause climate change which provides negative

Biofuel is

jatropha oil's demand and its market su

Calophyllum inophyllum L *Aleurites trisperma Blanco*

2. Methods

2.1. Materials

of 0.97 g/mL, 16.14%, and molecular weight of 884.8144 g/gmol was provided by Koperasi Jarak Lestari, Cilacap, Indonesia. Methanol (99 %v/v) and solid acid catalyst tin (II) chloride (99% purity) were obtained from Merck. The average molecular weight of the oil mixture was 884.2100 g/gmol.

2.2. Experimental Work of Esterification Reaction.

Prior to the esterification reaction, degumming process using 20% phosphoric acid was carried out to remove gum from the oils. Esterification reaction of the mixture of non-edible oils and methanol was then conducted in reactive distillation column as described in the previous work [6]. The reaction was performed at constant temperature of 60℃ and molar ratio of oil to methanol of 1:60. Catalyst employed in this work was tin (II) chloride with the amount of 5% w/w oil. Analysis of FFA conversion was performed using titrimetric analysis [7]. The initial oil mixture composition on the feed-stock was varied. Oil feed-stock composition variation was determined using Design Expert Software by applying Simplex Lattice Design Method. Based on this method, the different initial formulation of oil feed-stocks composition on 100 mL total mixture basis is depicted in Table 1. The feed-stocks formulation revealed the FFA content ranging from 8.42 to 12.26%.

Table 1. Experimental Design of Initial Oil Feed-Stock Composition

Run	CJO (mL)	CNO (mL)	WCO (mL)
1.	51.9	31.9	16.2
2.	27.5	50.0	22.5
3.	49.6	25.4	25.0
4.	70.5	10.0	19.5
5.	77.3	12.8	10.0
6.	40.0	50.0	10.0
7.	43.1	40.4	16.5
8.	35.0	40.0	25.0
9.	69.2	20.8	10.0
10.	59.9	15.1	25.0
11.	59.8	23.6	16.6

2.3. Optimization and Validation.

Optimization was carried out based on the data of reaction conversion of FFA (the decreasing of FFA content on the esterification reaction) on various oil feed-stocks composition. Simplex Lattice Design Method (SLDM) was applied for optimization process to determine the most appropriate composition of feedstock which provided the best FFA reduction through esterification reaction using reactive. Optimization was then validated by comparing the FFA reduction prediction using design expert software with the experimental data. The best composition was then used for biodiesel synthesis through transesterification reaction. Ethyl ester content in biodiesel produced from transesterification reaction was instrumentally determined using Gas Chromatography-Mass Spectroscopy (GC-MS).

3. Result and Discussion

3.1. Esterification Reaction

The FFA Esterification reaction of the non-edible oil mixture was accomplished based on the experimental design, in which the feed-stocks composition was determined using SLDM in Design Expert Software. The FFA conversion of each experiment was demonstrated in Figure 1. The FFA conversion achieved in this work was ranging from 70-85%, in which the FFA content could be reduced from 8.24-12.26% to 1-2%.

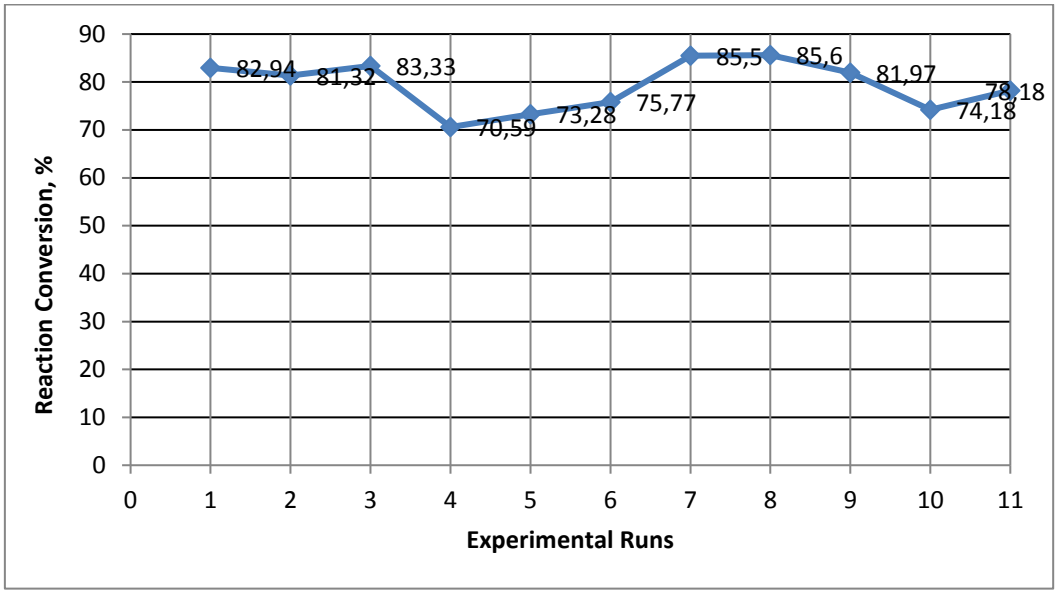

Figure 1. Conversion of Esterification Reaction at Reaction Temperature of 60℃, Ratio Molar of Oils to Methanol of 1:6, and Tin (II) Chloride Catalyst Concentration of 5% w/w oil for Various Feed-Stock Composition

3.2. *Response Analysis*

Each Response variable subsequently went through Analysis of Varians (ANOVA). The ANOVA model for variable analysis selected in this work was the model suggested by Design Expert Software 10.0, which provided highest level and significance. Based on ANOVA (Table 2), it was revealed that the selected model for each oil mixture respose was quadratic model. This model has shown the highest value of R^2 (0.944) compared to the other models. This model also demonstrated significance with the p value of p <0,0001 (<0,05). The ANOVA revealed that the CJO dan CNO provided significant effect on the response of FFA conversion. Lack of Fit (F-Value) was 4.46 (>0.05) indicating insignificant Lack of Fit. Insignificant value of Lack of Fit is a requirement for a good model since it indicates the suitability of the response data with the model [8].

Table 2. Method Analysis for the Response of FFA Reduction of Non-Edible Oil Mixtures

Response	Model	Significance (p<0.05)	Information
AB	Quadratic	0.7807	Insignificant
AC	Quadratic	0.0716	Insignificant
BC	Quadratic	<0.0001	Significant

A = Waste Cooking Oil (WCO)
B = Crude Jatropha Oil (CJO)
C = Crude Nyamplung Seed Oil (CNO)

Design Expert Software 10.0 also offers facility of normal plot residual which indicates whether the residual (the difference between the actual response and the predicted response value) fulfills the normal line/ straight line [9]. The residual plot was shown in Figure 2.

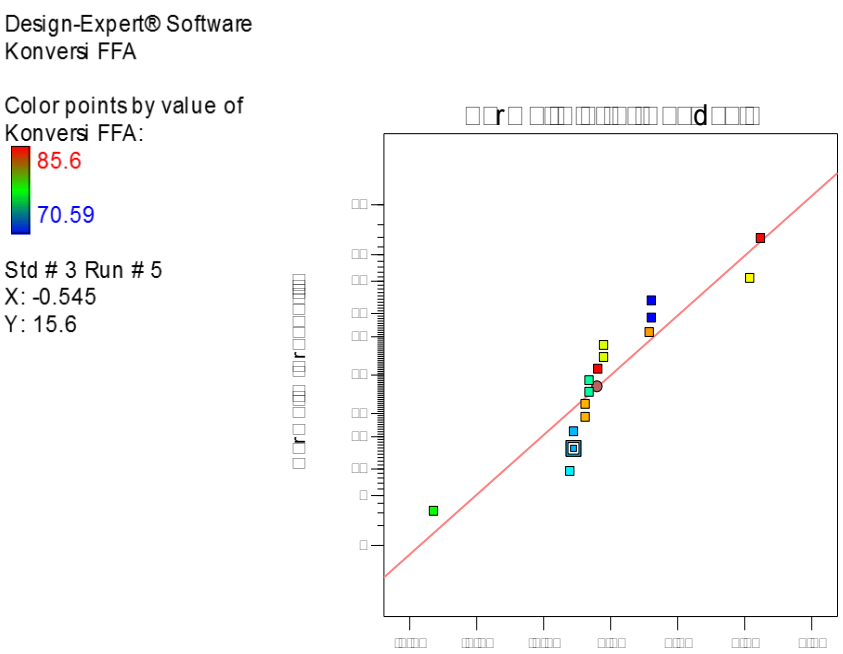

Figure 2. Residual Normality Response of FFA Reduction

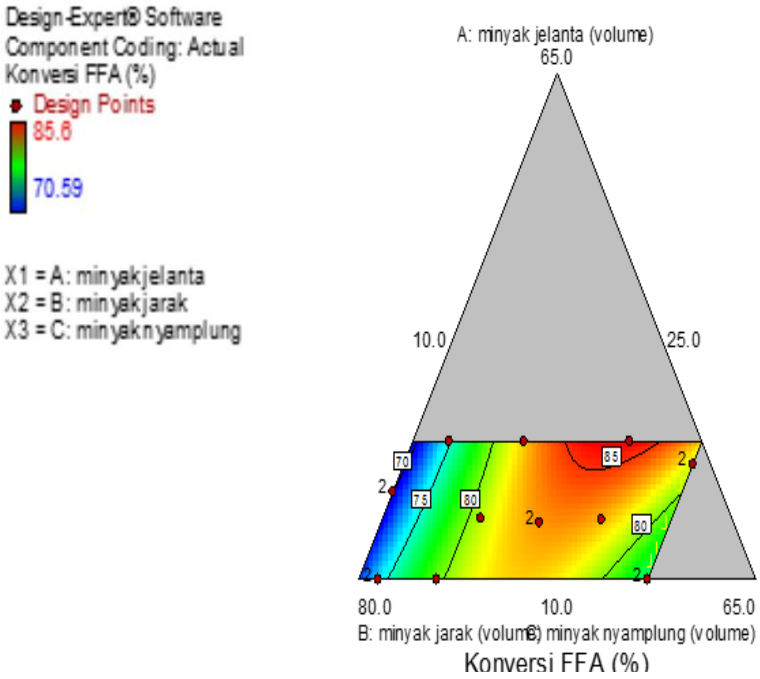

Figure 3. Contour Plot of Response Test of FFA Conversion

Contour plot in Figure 3 denotes how the combination of composition component affects the response of FFA conversion. The different colour on the contour plot specifies the value of FFA conversion. Blue colour indicates the response of the lowest conversion (70.59%). Red colour shows the response of the highest conversion (85.6%). The lines consisting of dots on the contour plot demonstrates the combination of the three components with the different composition which results in the similar yield. The surface shape of the interaction relationship between these components is depicted more clearly on the three-dimensional graph shown in Figure 4. The result of response analysis was then combined to obtain optimum composition of CJO, CNO, and WCO which yields the best FFA conversion.

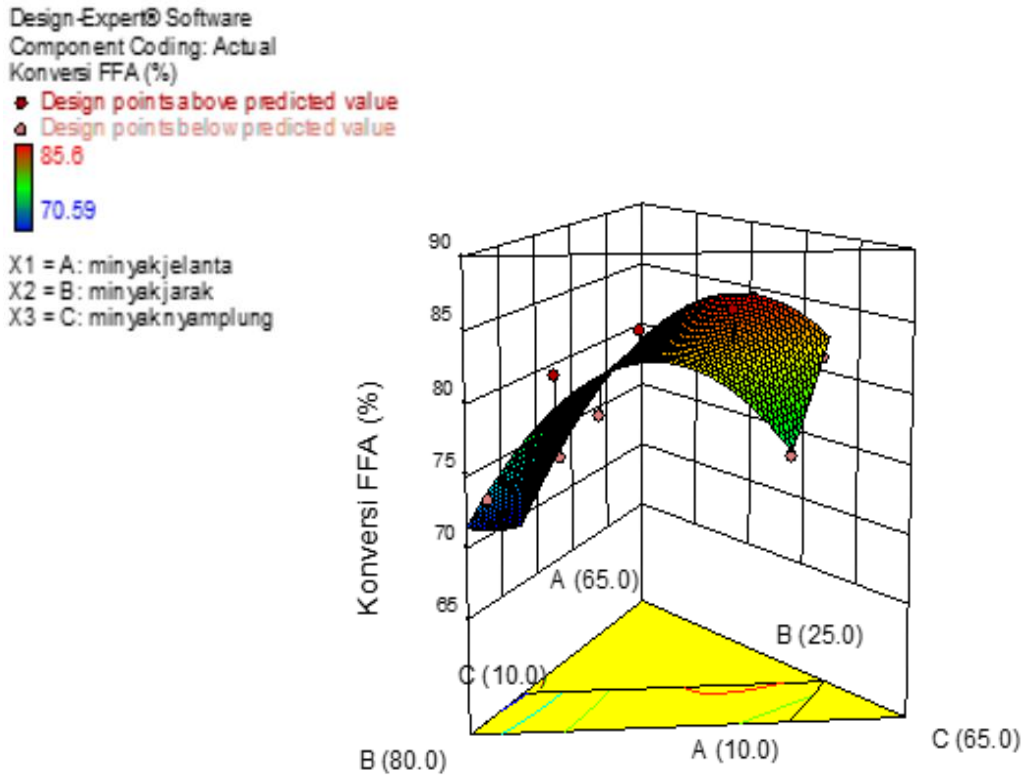

Figure 4. The Three Dimensional Response Test on the FFA Conversion

3.3. *Response Analysis*

Optimization was performed using Simplex Lattice Design Method (SLDM). This Method is able to carried out optimization according to the variable data and response measurement data inputted. Output of the optimization step is the recommendation of the new optimal formulation of the oil mixture. The optimal formulation is the composition which demonstrated the maximum desirability value. Desirability value is the function value for optimization purpose, indicating the ability of the program to fulfill the need based on the criteria specified on the final product.

In this work, optimization was conducted by determining the criteria (goal) of the desired FFA conversion which was ranging from 70.59% to 100%, thus the final FFA content resulted by the esterification reaction achieved the value as low as possible. Optimization using SLDM brought about the optimum FFA conversion of 85.938% with the formulation as shown in Table 3 and the desirability value of 0.937. The closer value of desirability to 1.0 denotes the reliability of the program to develop the desired result [11].

Table 3. Hasil Formula Optimum dari Metode *Simplex Lattice Design*

Composition	Prediction (mL)
CJO	37.291
CNO	37.709
WCO	25

The predicted value as shown in Table 3 was furthermore validated using experiment. Validation was carried out by evaluating the difference of the reaction conversion obtained by the predictive program and the empirical value obtained by experimental work for the similar non-edible oils feed-stock composition. The proximity between the predictive and empirical value indicates the validity of the model as demonstrated in Table 4.

Table 4. Comparison Between Predictive and Experimental Data of FFA Conversion Using the Optimal Composition of Feed-Stocks

Optimal Composition	Predictive FFA Conversion	Experimental FFA Conversion (Validation)
37.291 mL CJO + 37.709 mL NCO + 25 mL WCO	85.938%	86.03%

The validated optimum composition was then utilized as the feed-stocks for the transesterification reaction to produce biodiesel using reactive distillation process at the temperature of 60°C, molar ratio of oil to methanol of 1:6, and catalyst concentration of 1% NaOH catalyst [6]. Biodiesel produced contained 99.65% alkyl ester based on GC-MS analysis. The density of product was 884 kg/m^3, which meets the biodiesel standard.

4. Conclusion

Optimal composition of non-edible oil mixture which consists of crude jatropha oil, nyamplung seeds oil, and waste cooking oil can be predicted using Simplex Lattice Design Method (SLDM). The optimization resulted in the best composition of 37.291 mL CJO, 37,709 mL CNO, and 25 mL WCO which revealed in the optimum FFA conversion of 86.03%. Biodiesel produced after the oil mixture underwent transesterification reaction has the density of 884 kg/m^3 and alky ester content of 99.65%.

Acknowledgement

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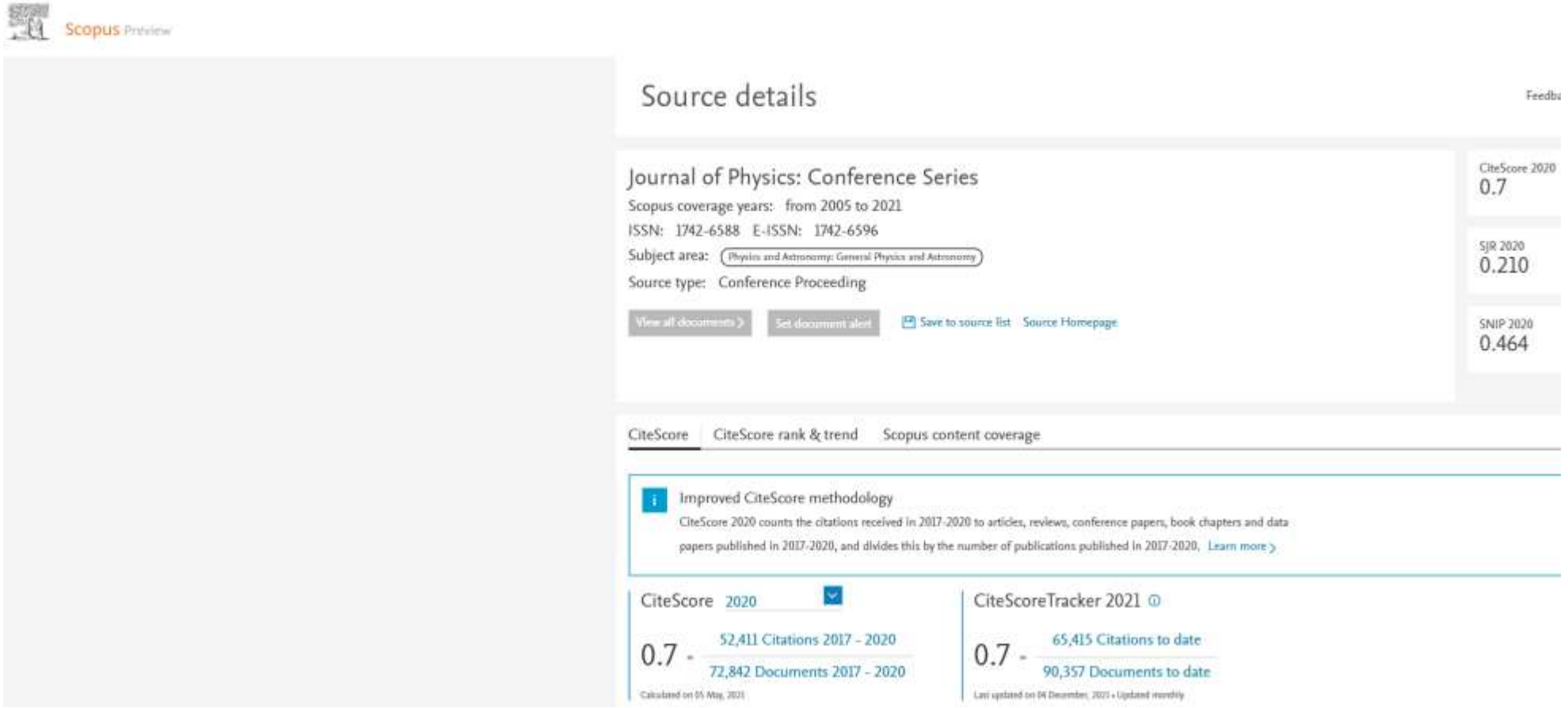

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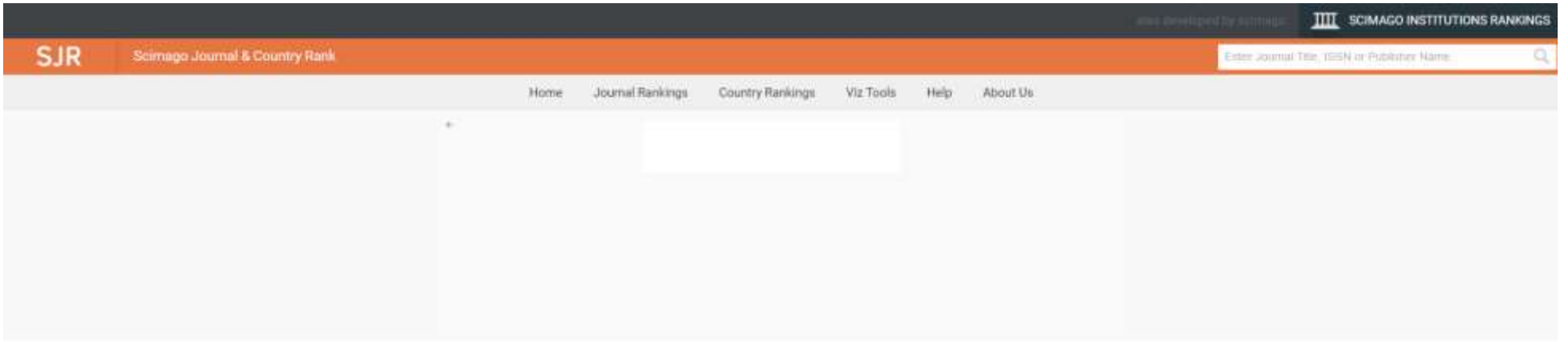

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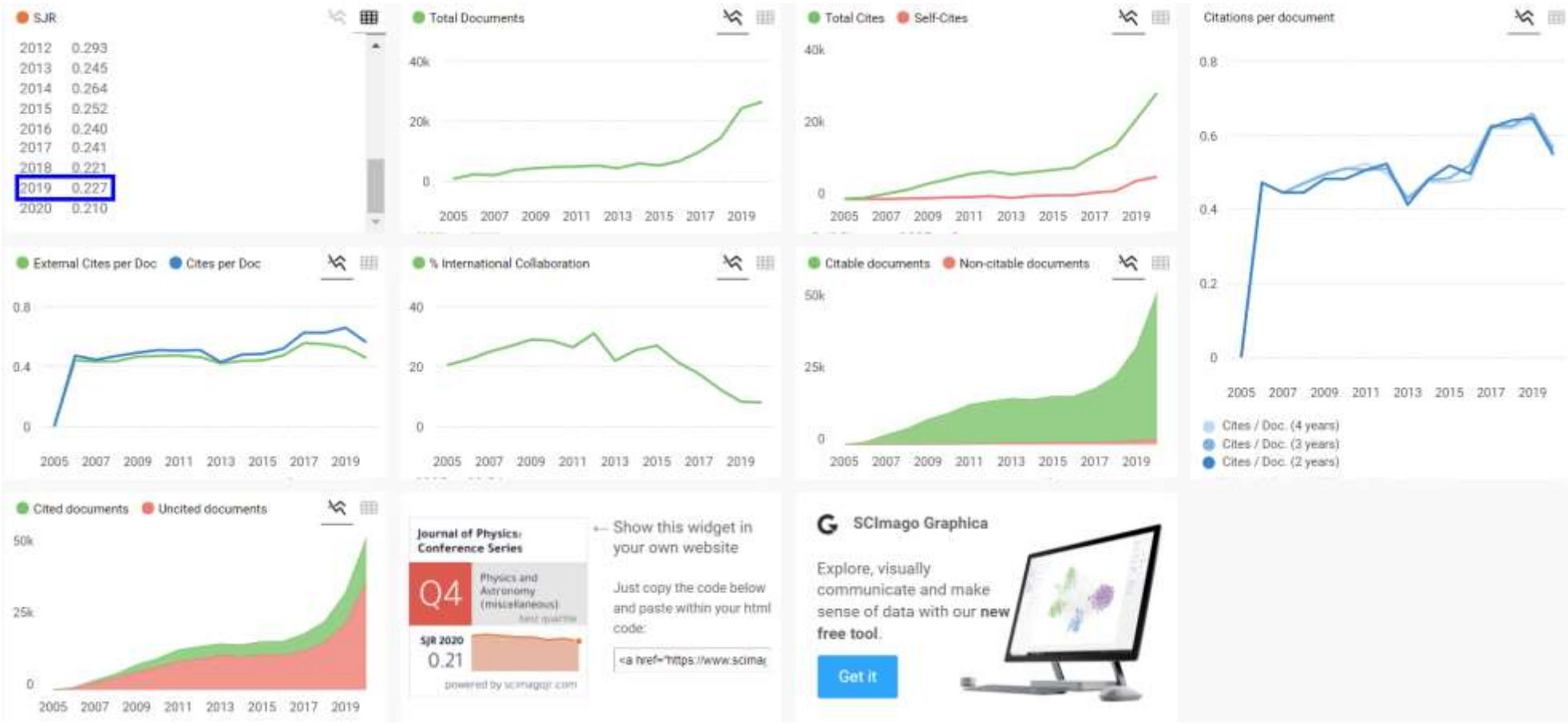

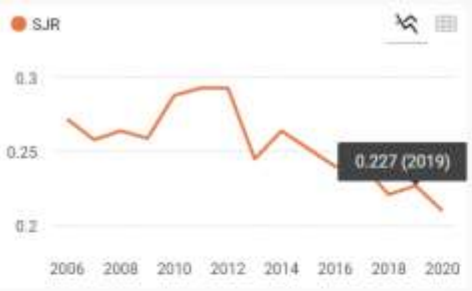
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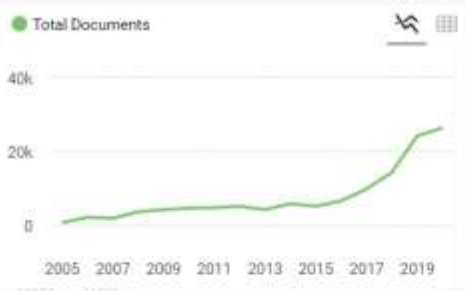
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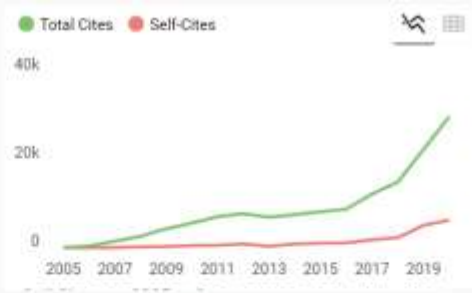
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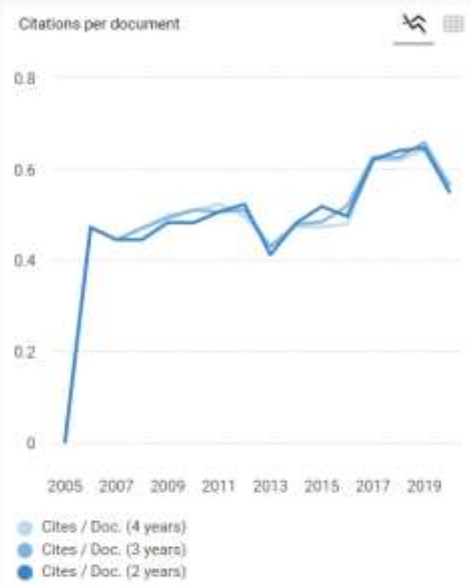
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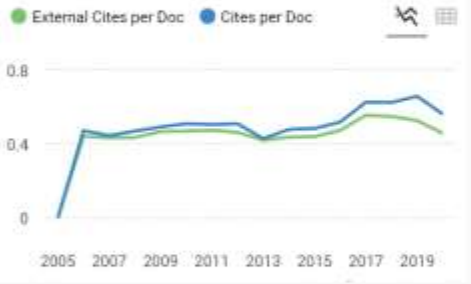
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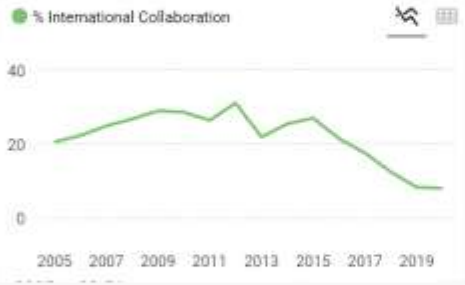
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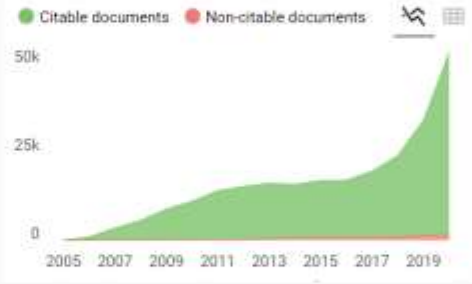
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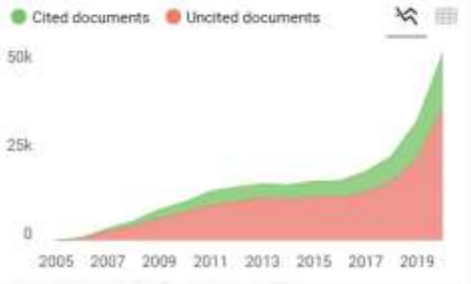
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