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Research article

Controlled crushing device-intensified direct biodiesel production of *Black Soldier Fly* larvae

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ABSTRACT

Insect larvae contain sufficient oil comparable with oleaginous biomass, and hence have potency as alternative biodiesel resources. The direct transesterification of Black Soldier Fly (BSF) larvae have conducted using a controllable crushing device (CCD) and a homogeneous base as a catalyst. The effect of catalyst concentration (wt.%), ratio BSF larvae to methanol (wt./v), reaction time (min) and rotational speed (rpm) on biodiesel conversion was determined. The maximum conversion of 93.8% was achieved at room temperature after 20 min of reaction time and ratio larvae to methanol of 1:2 (wt./v), catalyst concentration of 7 wt% and rotational speed of 3000 rpm. In addition, the green metrics calculation showed that this method produces less waste and uses less solvent. Some of the BSF-biodiesel properties meet the biodiesel standard. The CCD-intensified the DT of BSF larvae is a promising alternative for green and energy-saved biodiesel production.

1. Introduction

Research in biodiesel production has been switched to the utilization of non-edible biodiesel feedstock due to the food vs fuel problem [1,2]. Waste cooking oil is the best candidate as the price is lower than vegetable oil and the utilization is remaining limited [3,4]. However, the stocks are limited and require pre-treatment due to high free fatty acid content [5]. Insect larvae have potency as biodiesel feedstock as contain oil comparable to several vegetable oils [6,7]. Insect larvae of superworms, houseflies, meat flies, mealworm beetles and BSF have been explored and could produce biodiesel that meets the international standard requirement [8–13]. The BSF larvae potentially present an oil content amounting to 30–40% [6]. In addition, the BSF is harmless to human health as it does not spread germs [14]. The adult BSF will lay 500–900 eggs and it requires 4 days to hatch into larvae. At the larval stage, BSF consumes waste organic to grow [7].

The main bottleneck in current biodiesel production is lipid/oil preparation which accounted for 75–88% of total biodiesel cost [1,15,16]. Direct transesterification (DT) processes offer a simple biodiesel production through simultaneous extraction and transesterification of oil-bearing material [1,17]. In addition, the DT method eliminates the use of an air pollutant chemical in the extraction step with methanol acting both as an extraction solvent and a reactant [1,18,19]. Recently, the DT process has been explored either in a non-catalytic system [20] or using a catalyst conducted either in conventional reflux method [21] or using reaction-intensified devices

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such as microwave, ultrasound, and microfluidic [22–25]. Except for microfluidic device, all the DT process is conducted at high reaction temperature which is energy intensive [26]. Our previous study in the DT of rubber seed to biodiesel using the CCD showed that the biodiesel conversion of 97.5 ± 0.6% was achieved in 7 min at room temperature [16]. Therefore the present study demonstrates the DT of BSF larvae to produce biodiesel at room temperature. Based on the literature review, no work has investigated the use of CCD-intensified the DT of BSF larvae using a homogeneous catalyst.

This study reports the outcomes of the CCD-assisted the DT of BSF larvae operated in batch mode at ambient temperature and pressure using sodium hydroxide as a catalyst. The effect of processing parameters such as ratio BSF larvae to methanol, catalyst concentration (wt%), reaction time (minutes) and rotational speed (rpm) was systematically investigated to achieve maximum biodiesel conversion. Further, the biodiesel properties are predicted based on the fatty acid composition of the BSF larvae and compared with international standards. The green metric parameter such as environmental factor, process mass intensification, solvent intensity, wastewater intensity and effective mass yield also calculate to determine how green the process.

2. Materials and methods

2.1. Materials

The BSF larvae were bought from a local farmer roughly 3 days after hatching into larvae. The BSF larva was fed with coconut endosperm waste for roughly 10 days before being used as biodiesel raw material. All the chemicals used in this study were purchased from the local chemical distributor and were used as received.

2.2. Determination of BSF larvae oil content

The soxhlet method was used to quantify the oil content in the BSF larvae following previous procedures [16,27]. Firstly the BSF larvae were dried in an oven at 105 °C until constant weight and were ground with a coffee grinder. The dry biomass powder was placed in a thimble and extracted for 30 min using hexane as solvent. The solvent oil mixture was separated using the evaporation method and the BSF oil was weighted.

2.3. The DT of BSF larvae

The CCD mechanism work is adapted from a home capsule blender appliance equipped with a controller to adjust the rotational speed. There are two blades attached to the shaft connected to the motor. Those blades work both to disrupt the BSF larvae and to increase mass transfer between the reactants. The parameters tested in this study were catalyst concentration of 0.5; 1; 3; 5; 7; 9 and 12 wt%, a ratio of BSF larvae to methanol of 1:1; 1:2; 1:3; 1:4 and 1:5 (wt./v), a reaction time of 10, 20, 30, 40, 50, 60, 75 and 90 min, and rotational speed of 3000–8000 rpm with an increment of 1000 rpm. The CCD-intensified the DT of BSF larvae was carried out in a glass reactor. A 10 g of BSF larvae was stirred with a prescribed concentration of catalyst and methanol. Next, the reactants were agitated with assigned rotational speed and reaction time. The biodiesel product was then separated from residual larvae using a vacuum filter followed by separation in a separation funnel to separate leftover methanol and glycerol as a by-product. The biodiesel was washed with warm water, dried and stored in a desiccator for conversion analysis.

2.4. Biodiesel conversion determination

The biodiesel conversion is determined using gas chromatography Shimadzu type 2010. The GC is equipped with a capillary column (length 30 m, ID 0.25 mm and film thickness of 0.25 μm) and flame ionization detector. The biodiesel sample (1 μL) was injected into the injection port (temperature set to 260 °C) with helium as a gas carrier (flow of 30 mL/min). The biodiesel conversion was calculated based on equation (1) with methyl heptadecanoate as the internal standard.

$$Biodiesel\ Conversion\ (\%) = \frac{\sum A - A_{IS}}{A_{IS}}\ x\ \frac{C_{IS}V_{IS}}{m} x\ 100 \tag{1}$$

where $\sum A$ are total peak area of fatty acid methyl ester, A_{IS}, C_{IS} and V_{IS} are peak area, concentration (mg/mL) and volume internal standard (mL), respectively while m is the mass of the methyl ester sample.

2.5. Biodiesel properties prediction

The biodiesel properties could be predicted based on the fatty acid composition of the obtained fatty acid methyl ester. The physicochemical properties of cetane number, density, viscosity, oxidative stability, pour point and cloud point were calculated based on the equations shown in some previously published reports [16,28,29]. The detailed equations to calculate those properties were listed in the supplementary information.

2.6. Green metrics calculation

To determine how green the CCD-intensified the DT of BSF-larvae process is, the green metrics equations were adopted from a previously published report [30] and used to calculate the environmental impact of this study.

$$E-factor=\frac{Total\ mass\ of\ waste}{Mass\ of\ biodiesell\ produced} \tag{2}$$

$$Process\ mass\ intensification=\frac{Total\ mass\ in\ process}{Mass\ of\ biodiesel\ produced} \tag{3}$$

$$Solvent\ Intensity=\frac{Mass\ of\ solvents}{Mass\ of\ biodiesel\ produced} \tag{4}$$

$$Waste\ water\ intensity=\frac{Mass\ of\ process\ water}{Mass\ of\ biodiesel\ produced} \tag{5}$$

$$Effective\ mass\ yield=\frac{Mass\ of\ biodiesel\ produced}{Mass\ of\ hazardous\ reactants} \tag{6}$$

2.7. Statistical analysis

The one-way analysis of variance (ANOVA) was used to determine the significant effect of parameters using Statistica v13 with a significance level set to $\alpha = 0.05$. The Tukey post hoc test is used to compare the means.

3. Results and discussion

The oil content, organic substrate and fatty acid composition of the lipids from BSF larvae of this study and other published results are shown in Table 1. The oil extracted from BSF larvae was 38.5% of dry larvae. The result is in the range of oil content of 10–58% [12, 31] It worth noting that oil content is highly dependent on the organic substrate type. Mohan, Sathishkumar [31] summarise the yield of lipids of BSF larvae from the different organic substrates and concluded that the diet type affects the lipid content. The fatty acid composition of the lipids from BSF larvae confirms the feasibility of this insect as a biodiesel and was found to be similar to other reported data [12,31–34]. The saturated fatty acids of this study dominated the oil content at 67% while monounsaturated and polyunsaturated fatty acids were 21.6 and 11%, respectively. Lauric and palmitic acid are the major component of the saturated fatty acids contents which occupy 55.8%. The high amounts of medium-chain fatty acids provide better oxidative stability of biodiesel [35].

3.1. Effect of catalyst concentration

The reaction condition of the ratio BSF larvae to methanol of 1:2 (w/v), a reaction time of 30 min and a rotational speed of 5000 rpm was used to investigate the influence of catalyst concentration. The reaction condition used in this study was opted based on the previously published result [16]. Fig. 1A shows the effect of catalyst concentration on biodiesel conversion. The catalyst had a

Table 1
Comparison oil content, organic substrate and FA composition of some BSF larvae.

Oil Content/Organic Substrate/Fatty Acid		Percentage				
		This Study	[32]	[12]	[33]	[34]
Oil content		38.5%	24.7%	36.2%	49.4%	39.6%
Organic substrate		Coconut endosperm waste	Palm kernel expeller	Food waste	Coconut endosperm waste	Rice straw & Restaurant waste
$C_{10:0}$	Capric	1.1	1.2	2.1	1.3	3.8
$C_{12:0}$	Lauric	35.3	47.5	47.4	66.0	27.8
$C_{14:0}$	Myristic	7.8	14.4	8.2	14.4	8.1
$C_{14:1}$	Myristoleic	n.d.	n.d.	n.d.	1.4	n.a.
$C_{16:0}$	Palmitic	20.5	14.6	14.4	6.6	14.2
$C_{16:1}$	Palmitoleic	2.8	3.1	2.4	2.2	4.5
$C_{18:0}$	Stearic	2.2	2.1	2.8	0.4	7.6
$C_{18:1}$	Oleic	18.8	11.1	16.3	5.6	22.5
$C_{18:2}$	Linoleic	10.1	4.0	6.3	2.3	1.8
$C_{18:3}$	Linolenic	0.9	n.d.	n.d.	n.a	2.1
$C_{20:0}$	Arachidic	0.1	n.d.	n.d.	n.d.	n.d.
$C_{22:1}$	Euric	n.d.	2.0	n.d.	n.d.	n.d.
SFA		67.0	79.8	74.9	88.7	61.5
MUFA		21.6	16.2	18.7	9.2	27.0
PUFA		11.0	4.0	6.3	2.3	3.9

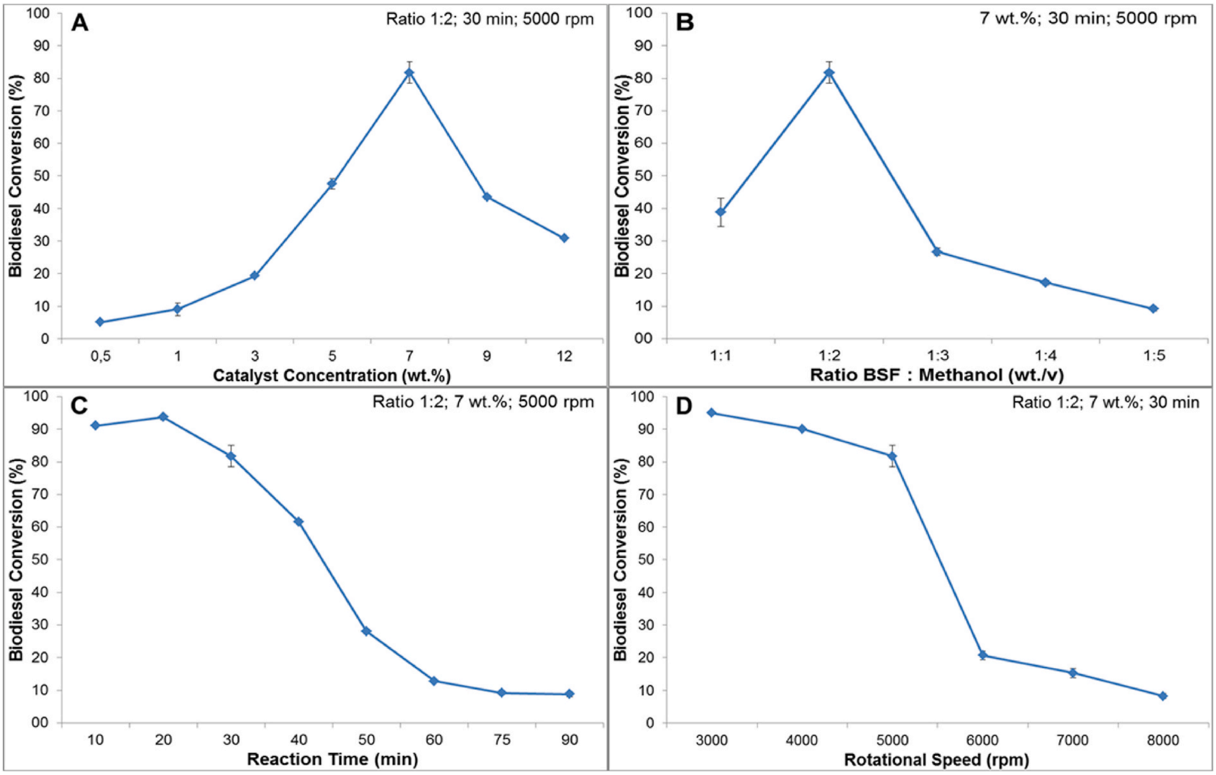

Fig. 1. The effect of (A) catalyst concentration; (B) ratio BSF: methanol; (C) reaction time; and (D) rotational speed on biodiesel conversion.

significant effect on the biodiesel conversion as the increment of catalyst concentration up to 7 wt% raised the conversion. This presumably due to increasing concentration would increase the active sites of the catalyst involved in the transesterification reaction [1]. However, as the catalyst concentration was increased, the conversion decreased sharply from 81.8 ± 3.3% at 7 wt% to 43.5 ± 0.7% at 9 wt%. This result is due to saponification emerging in an excess concentration of sodium hydroxide [16,36]. A similar result was also observed in our previous study on the DT of waste rubber seed using a similar device [16]. Taherkhani and Sadrameli [37] also obtained a similar result in the DT of linseed using tetrahydrofuran as a co-solvent and potassium hydroxide as a catalyst. Their result showed that the biodiesel yield decreased after reaching the optimum point in the increase of catalyst concentration [37]. Statistical analysis was performed using ANOVA and the result indicates that catalyst concentration has a significant effect on biodiesel conversion. The Tukey post hoc further showed that all concentrations were significantly different.

3.2. *Effect of ratio BSF larvae to methanol*

Methanol is acted both as a solvent and a reactant in the DT processes [1]. Therefore an excessive volume of methanol, which is exceeded the stoichiometric molar ratio of triglycerides to methanol, is necessary to ascertain complete extraction and transesterification reaction [17]. Hence, the ratio of BSF larvae to methanol (wt./v) in the range of 1:1 to 1:5 was investigated under reaction conditions of catalyst concentration of 7 wt%, a reaction time of 30 min and a rotational speed of 5000 rpm. The ratio of 1:1 was chosen as it is a minimum volume of methanol to immerse the larvae. Firstly, a low conversion of 38.8 ± 4.4% was achieved when a ratio of 1:1 (wt./v) was employed and it was increased to reach a maximum conversion of 81.8 ± 3.3% at ratio of 1:2 (wt./v). Further increase in the methanol volume, the biodiesel conversion was observed decreased to 9.2 ± 0.6% at ratio of 1:5 (Fig. 1B). This presumably due to low volume of methanol is insufficient to extract the oil while an excessive volume of methanol dilutes the larvae and catalyst concentration leading to a reduction in biodiesel conversion [31,37]. This result is consistent with the findings of Nguyen, Nguyen [38] who concluded that the biodiesel yield increases up to the peak as the ratio increases before further down as the surplus of methanol dilutes the switchable solvent [38]. The ANOVA results showed that the ratio of BSF larvae to methanol had a significant effect on biodiesel conversion which was driven by the low conversion at a ratio of 1:5.

3.3. *Effect of reaction time*

In the CCD-intensified the DT of BSF larvae, reaction time is an important factor since it provides the reaction mixtures sufficient contact time under high rotation speed resulting in better extraction and transesterification reaction. In response to this, the reaction condition of ratio BSF larvae to methanol of 1:2, catalyst concentration of 7 wt% and rotational speed of 5000 rpm was used to determine the effect of reaction time. The biodiesel conversion of >90% was achieved for a reaction time of 10 and 20 min as shown in Fig. 1C. However, when the reaction time was extended to more than 20 min, the biodiesel conversion was decreased. The conversion decreased from 93.7 ± 0.8% at a reaction time of 20 min–81.8 ± 3.3% and 8.8 ± 0.8% at reaction times of 30 and 90 min, respectively. This phenomenon also occurred in the DT of waste rubber seed using CCD. The conversion in those studies decreased from 97.5 ± 0.6% at 7 min to 62.9 ± 0.5% at 15 min [16]. Transesterification is a reversible reaction, hence prolonged reaction time can favour the backward reaction resulting reducing in biodiesel conversion [39]. Laskar, Gupta [40] in the study of the utilization waste *Mangifera indica* peel as a heterogeneous catalyst in biodiesel production from soybean oil concluded that hydrolysis of the methyl ester may occur in the prolonged reaction time [40]. Further, the results were analyzed using one-way ANOVA to determine the significant effect. The reaction time has a significant effect on biodiesel conversion.

3.4. Effect of rotational speed

Agitation is a crucial parameter that affects the mixing degree of reactants and mass transfer. Diffusion between reactants is increased with the increasing mixing intensity yielding an increasing reaction rate [16,17]. In this investigation, rotational speed was considered in the range of 3000–6000 rpm with an increment of 1000 rpm. To determine this parameter, a reaction condition of ratio BSF larvae to methanol of 1:2, catalyst concentration of 7 wt% and reaction time of 20 min was applied. Interestingly, the maximum biodiesel conversion of 95.0 ± 0.3% was achieved using the lowest rotational speed (3000 rpm) as shown in Fig. 1D. Further in increasing rotational speed, the conversion was decreased with the minimum conversion of 8.2 ± 0.8% obtained when a rotational speed of 8000 rpm was applied. This result revealed that a rotational speed of 3000 rpm was sufficient for both extraction and transesterification of BSF larvae to biodiesel. Kasirajan [41] also obtained a similar result in the transesterification of *Chrysophyllum albidum* oil. In this context, Joshi, Gogate [26] concluded that in high rotational speed the cavities could be collapsed due to super cavitation produced [26]. One-way ANOVA revealed a significant interaction between rotational speed and biodiesel conversion which was driven by all the values tested.

3.5. Comparison of the different methods for the DT of BSF larvae

Table 2 summarizes the reaction conditions and outcomes achieved for the DT of BSF larvae to biodiesel. All of the DT studies showed a biodiesel conversion of >93% with the highest conversion of 96.2% achieved by Nguyen, Nguyen [38]. Most of the studies used catalysts either homogeneous acids or base substances or switchable solvents except for the study by Jung, Jung [12] who used the thermal induced method. Even though the non-catalytic DT method achieved a biodiesel conversion of 94.1% in 1 min reaction time, the reaction temperature is very high (390 °C). However, this method is energy intensive and thermal decomposition of the product could occur in prolonged reaction time [42,43]. Except for this study which was performed at room temperature, other studies which use the reflux method were also conducted at high reaction temperatures (120 and 110 °C). The ratio of BSF larvae to methanol used varied from 1:2 to 1:16 (wt/v) where this study uses fewer methanol than others.

3.6. Biodiesel properties

It is well known that the physicochemical properties of biodiesel are highly related to the fatty acid composition contained in its fatty acid methyl ester [16,28,29]. The biodiesel properties of cetane number, density, viscosity, oxidative stability, pour point and cloud point of fatty acid methyl ester from some insect larvae and palm oil are calculated and presented in Table 3. The biodiesel obtained from BSF larvae met the international standard regulation except for ester content and viscosity. The ester content could be increased using another type of catalyst and intensified devices such as microwave and ultrasound. Low viscosity is presumably due to the high concentration of medium-chain fatty acid contained in BSF-biodiesel [35]. However, the cetane number of BSF-biodiesel was higher than the minimum standard of EN 14214 and ASTM D6751 and comparable with biodiesel from palm oil. The higher cetane number of BSF-biodiesel means a shorten delay time for diesel machines to ignite [17]. The higher content of saturated fatty acid in biodiesel has positive and negative impacts on its properties. The oxidation stability increased in the increasing saturated fatty acid while the cold flow-temperature properties could be decreased [17,45]. The oxidation stability of BSF-biodiesel is slightly higher than other insect larvae biodiesel but lower than palm oil biodiesel. However the cloud and pour point of BSF-biodiesel were slightly lower

Table 2

Summary of a different methods of the DT of BSF larvae to biodiesel.

Method	Type of Insect and Reaction Condition	Biodiesel Conversion/ Yield	Ref.
Reflux	BSF; ratio hexane: methanol of 1:2 (v/v); ratio BSF larvae: solvent of 1:6; H_2SO_4 loading of 1.2 mL; 120 °C and 90 min.	94.14%	[44]
Reflux	BSF; ratio BSF larvae: methanol of 1:8 (wt/v); ratio BSF larvae to catalyst of 1:16 (wt/v); 110 °C and 60 min.	96.2%	[38]
CCD	BSF; ratio BSF larvae: methanol of 1:2 (wt/v); NaOH concentration of 7 wt%; room temperature and 20 min.	93.8%	This study
Thermal induced	BSF; ratio BSF larvae: methanol of 1:10 (w/w), 390 °C and 1 min.	94.1%	[12]

Table 3
Fuel properties of biodiesel from insects' larvae and palm oil.

Properties	Units	EN 14214	ASTM D6751	BSF	Yellow mealworm	Zophobas morio	Housefly	Palm Oil
Ester content	%	>96.5	–	93.8	n.a.	92.4	90.3	98
Cetane number	–	>51	>47	60	51	57	57	60
Density	25 °C, kg m^{-3}	860–900	–	869	877	853	853	858
Viscosity	40 °C, mm^2 s^{-1}	3.5–5.0	1.9–6.0	3.0	3.7	3.7	3.5	3.9
Oxidative stability	H	8	>3	13.3	6.2	7.6	9	19
Pour point	°C	Report	Report	−0.5	−1.8	6.5	4.4	13.1
Cloud point	°C	Report	Report	5.8	4.6	12.2	10.3	7.4
References				This study	[20]	[8]	[46]	[47]

than palm oil-biodiesel.

3.7. *Green process metrics and processing parameter comparison*

The demand to determine how green the chemical process is soared currently due to environmental concerns [30,48]. The chemical process which generates less waste and avoids the use of hazardous chemicals is classified as green chemistry. The green metrics process such as environmental factor, process mass intensity, solvent intensity, reaction mass efficiency, mass productivity and effective mass yield were calculated in this study using equations (2)–(6) and compared with other DT processes [38]. Due to insufficient data for other DTs of BSF larvae, a comparison was made using the data from the DT of yellow mealworm larvae [20]. Table 4 showed the mass reactants used in the DT of BSF and yellow mealworm larvae using CCD-intensified, conventional reflux and supercritical methanol methods.

The e-factor which determines the ratio of waste product per biodiesel yield showed that the CCD-intensified the DT of BSF larvae have less than 87.6% of the waste product than the conventional reflux method but produce 60% more waste than the supercritical methanol method (Table 5). This is understandable considering the supercritical methanol method did not use organic solvent, catalyst and water for purification hence less waste product was produced. As shown in Table 5, the CCD-intensified the DT of BSF larvae performed better in the process of mass intensification and solvent intensity than the other DT methods. The conventional reflux and supercritical methanol methods require an excessive volume of methanol to perform the DT reaction [49] which therefore increases the solvent intensity. However, due to the use of water for the neutralization of the catalyst and purification of the product, the CCD-intensified the DT of BSF larvae method had a higher wastewater intensity than the supercritical methanol method. Furthermore, the mass production of this study was 93% and 87% superior to conventional reflux and supercritical methanol methods, respectively. In terms of effective mass yield, the CCD-intensified the DT of BSF larvae was 84% and 89% more effective than other DT methods.

Processing parameters such as reaction time and temperature are the important factors that directly affect either biodiesel yield or production cost [1,17]. The reaction time in the supercritical methanol process is more reactive compared to CCD-intensified and conventional reflux methods. This could be attributed to that at critical points methanol easily disrupts the cell walls of insect larvae dissolving oil-producing biodiesel at a short reaction time [50]. However, the critical point is only achieved under high temperatures and pressure. The CCD-intensified the DT of BSF larvae is performed at room temperature which consumes less energy than conventional reflux and supercritical methanol methods.

4. Conclusion

This work demonstrated the CCD-intensified the DT of BSF larvae to biodiesel using a homogenous base catalyst at room temperature. The maximum biodiesel conversion of 93.8% was achieved under the reaction condition of ratio BSF larvae to methanol of 1:2 (wt./v), catalyst concentration of 7 wt%, a reaction time of 20 min and rotational speed of 3000 rpm. The predicted BSF-biodiesel properties based on fatty acid composition met the international standard requirement except for ester content and viscosity. In terms of green metrics, the CCD-intensified the DT of BSF larvae could be classified as green as produced less waste and used less chemical solvent. In addition, this method is performed at room temperature which consumes less energy.

Author contribution

Eko K. Sitepu: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

Sabarmin Perangin-angin, Gloria J. Ginting: Performed the experiments; Analyzed and interpreted the data.

Siti Machmudah, Rodiah N. Sari: Contributed reagents, materials, analysis tools or data; Wrote the paper. Juliati Br. Tarigan: Performed the experiments; Wrote the paper.

Data availability statement

Data included in article/supp. material/referenced in article.

Table 4
The mass of reactants used in the DT of insect larvae using different methods.

Reactants (g)	CCD-Intensified	Conventional Reflux	Supercritical Methanol
Species	BSF Larvae	BSF Larvae	Yellow Mealworm Larvae
Biomass	20,00	1,00	0,02
Biomass residue	12,04	0,75	0,0161
n-Hexane	0,00	0,00	0,00
Oil	7,70	0,25	0,0048
Catalyst	2,80	16,00	0,00
Water	60,00	3,00	0,00
Methanol	40,00	8,00	0,2
Fatty Acid Methyl Esters	7,32	0,24	0,0039
Reaction time (min)	20	110	1
Reaction temperature (°C)	RT	60	370
References	This Study	[38]	[20]

Table 5
Green metrics of different DT methods of insect larvae.

Green Metrics	CCD-Intensified	Conventional Reflux	Supercritical Methanol
E-factor (g/g)	10,22	82,29	4,15
Process mass intensity (g/g)	16.78	116.67	56.41
Solvent intensity (g/g)	5.46	33.33	51,28
Wastewater intensity	7.79	12.00	0
Mass productivity (%)	14.50	0.99	1.90
Effective mass yield (%)	18.30	3.00	1.95

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.heliyon.2023.e16402.

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