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Modeling and Optimization of Biodiesel Production from Croton macrostachyus Leaves Oil

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Abstract

The biodiesel produced from Croton macrostachyus (CM) leaves mostly contains unsaturated fatty acid esters with low stability of oxidation. A Croton macrostachyus (CM) leaf, a non-edible resource, was utilized to produce biodiesel. This novel work focuses on the trans-esterification of species known as CM leaves oil to produce biodiesel with the help of CaO nanoparticle (CaO NPs)-catalyzed technique. The esterification process is optimized utilizing response surface methodology (RSM) based on central composite design (CCD). Four parameters that affect the production of biodiesel from Croton macrostachyus (CM) leaves oil have been examined. The optimum operating conditions for the selected four factors have been investigated as reaction time 25.95 min, temperature 63.325 °C, methanol to oil ratio 28.093:1 in mg/L, and catalyst concentration 3.001% wt with a desirability value of 1. Under the predicted parameters, to optimize the production of biodiesel, the quadratic mathematical models were developed. The optimized trans-esterification result showed that a 96.375% yield of biodiesel (FAME) was found. Three different experimental runs were carried out to validate the proposed model by using the optimized process parameters, and 95.818% (average) of experimental yield have been found. The CM leaves oil biodiesel physicochemical properties were obtained, and it was observed all the tasted properties agree with fuel specifications set by ASTM D6751 standards. In conclusion, this work formulates the baseline and the need for future exploration of CM leaves oil for biodiesel production through different methods.

Keywords Croton macrostachyus leaves \cdot Biodiesel \cdot Optimization \cdot RSM \cdot Central composite design

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Introduction

Nowadays, climate change is a challenging problem, and the global community's primary energy sources are highly related to nonrenewable and unsustainable fossils. A day-by-day use of fossil increases greenhouse gas (GHG) production and rising global warming [13]. Many researchers addressed that if use of fossil as primary sources of energy continue for the next two decades, petro-diesel can be out of stock. Also limited resources, geopolitical disturbances, and destructive global effects of petro-diesel energy necessitates the search for an alternative renewable energy source [25]. One of the major alternatives to minimize or reduce such problem is the use of biodiesel fuel as an alternative fuel. Biodiesels have combustion property similar to petro-diesel, which can be produced from locally available biomass feedstock and can be used in the recent engine technologies without major amendments [8]. Additionally, the use of biodiesel minimizes emissions of global warming as it contains insignificant quantity of carbon and sulfur [17, 21].

Different feedstocks including seed oils, animal fats, herbal oils, vegetable oils, and waste cooking oils have been utilized for the production of biodiesel [10]. More than 355 oil companies can be recognized as a source for biodiesel production [5]. These can be grouped under two major classifications: non-edible and edible oils. Non-edible oils are the better candidate as cheap cost sources to produce biodiesel. For example, among the non-edible oil, *Croton macrostachyus* (CM) leaves oil can be used to produce biodiesel.

Chemically, biodiesel is a renewable energy that can be defined as fatty acid methyl esters (FAME) and it is a mixture of unsaturated and saturated fatty acids that are produced from the reaction between triglycerides and alcohol through trans-esterification process with the help of catalysts (heterogeneous or homogeneous) [12, 28].

Several works have been done on potential feedstocks and essential activities for improving the production of biofuels using the second-generation feedstock [9, 18, 22, 24]. The main reasons are that these second-generation feedstocks are available as low-cost sources. For example, the *Croton macrostachyus* (*CM*) *leaves* are abundantly available in Ethiopia [1]. *Croton macrostachyus* has been normally planted as shade trees. Like any other oils, *Croton macrostachyus* leaves oil conversion to biodiesel can be carried out through trans-esterification process. Trans-esterification process is a reaction between fat and oil with alcohol to produce esters and glycerol with the activity of catalysts (alkali or enzymatic) to improve the yield and reaction rate.

Biodiesel processed through trans-esterification process is a proficient approach breaking down the oils [3]. Production of biodiesel through trans-esterification helps to reduce the viscosity of oils to give efficient diesel. Arumugam et al. [3] studied an optimizing biodiesel production from *Calophyllum inophyllum* seed oil by utilizing calcined ash from leaf of sugarcane as a catalyst. Rezania et al. (2022) studied the production of biodiesel from wild mustard (*Sinapis arvensis*) seed oil with the help of LaTiO₃ nanoparticles as a catalyst by considering the processing operation conditions as reaction time, molar ratio of alcohol to oil, catalyst concentration, and temperature. They have optimized these conditions by using RSM methodology.

Response surface methodology (RSM) can be defined as an approach that has been used to optimize and predict the conditions of process operations, and it can explore the influences of various independent factors and their combined interaction on the response values [15, 18, 26, 27].

Response surface methodology can be used to perform various experimental designs such as oil extraction from plants and production of biodiesel from different feedstock to estimate their optimum operating conditions [7, 19, 20]. Ideris et al. [15] applied RSM based on Box-Behnken experimental design (BBD) to find the optimum operating conditions such as level of ultrasound amplitude, temperature time, n-hexane to solid molar ratio powder for the extraction of oil from COK via ultrasound-assisted method. Matthew et al., [20] applied both RSM and artificial neural network (ANN) to optimize the operating conditions such as temperature, extraction time, and solvent to oil ratio for the extraction of oil from *Terminalia catappa* L. kernel.

Recently, Girma et al. (2022) applied the tool of RSM based on CCD to determine the optimum operating conditions for the extraction of oil from *CM* leaves. All these studies have shown that the main idea of RSM is to use a set of advanced designed experiments to determine an optimum response value and make sure the efficiency of the model to save time and costs required in performing the experiment.

To the best of the authors' knowledge, no finite report has been addressed on the production of biodiesel from *CM leaves* oil. Therefore, this study is the first attempt, and modeling and optimizing biodiesel production from *Croton macrostachyus leaves* oil were studied in the presences of CaO nanoparticle catalyst. Then, optimum parameters will be identified with the help of RSM methodology based on CCD. The operating parameters such as molar ratio of methanol to oil, temperature, reaction time, and amount of catalyst are the major factors that are considered to obtain the optimum biodiesel yield from *Croton macrostachyus leaves* oil. The produced biodiesel oil properties are measured using ASTM D6751 test technique to make sure that their compliance with set standards.

Methodology

Extraction and Properties of CM Leaves Oil

CM leaves were collected from nearby Jimma Institute of Technology, Jimma City, Ethiopia. The leaves were air-dried in a shaded area for 2 weeks, and then, the air-dried leaves were powdered using a mechanical hand grinder to have high surface area and fine and uniform particles. Then, the powdered leaf (600 g) was kept into Soxhlet in which n-hexane was added and extraction time varied 1-2 h. The extracted oil has been separated from the solvent with the help of a vacuum rotary evaporator below 50 °C.

The total molecular weight (various fats and oils) of *Croton macrostachyus oil* extracted from leaf was determined by using the following formula mentioned in Eq. (1).

Average molecular weight of fatty acids =
$$\frac{\sum f_i}{\sum(\frac{f_i}{MW_i})}$$
 (1)

Croton macrostachyus oil has various compositions of fats and oil such as linolenic acid, oleic acid, palmitic acid, linoleic acid, and stearic acid. The physiochemical properties of *Croton macrostachyus leaf oil* such as oil density, kinetic viscosity, saponification value refractive index, iodine value, peroxide value, free fatty acids, and acid value were characterized.

Trans-esterification Process (Production of Biodiesel)

Trans-esterification process is a reaction between the vegetable oil (triglyceride) and alcohol in the presence of catalysts resulting with production of fatty acids alkyl esters and glycerol. Catalysts are used to improve the reaction rate and ester production. Since the trans-esterification process is a reversible process, more ration of alcohol is utilized favoring the forward reaction to produce more esters. The produced byproduct glycerol is removed, and the ester is purified using warm water into biodiesel.

In this work, the trans-esterification of *Croton macrostachyus oil* is carried out. There are several factors (i.e., amount of alcohol, temperature of reaction, the oil FFA content, time, and catalyst type and concentration) in the trans-esterification reaction that affects yield and quality of biodiesel.

Since the percentage of free fatty acids (FFA) of *Croton macrostachyus (CM) leaves* oil is much less when compared to the suggested FFA contents of the oil of vegetables for nano-catalytic trans-esterification, the trans-esterification of *Croton* \macrostachyus (CM) oil does not need a pretreatment technique or no esterification process [16]. Nano-catalyzed trans-esterification is processed in a 500-mL flask reactor, and 25 g oil was used for each nano-catalyzed trans-esterification experiments.

The reaction was allowed to take place for 50 min, and after the reaction is completed, the final product was kept in a separate funnel and left for one night for the separation purpose. Two layers were observed, bottom layer containing glycerol and other impurities and the upper layer consisting of the biodiesel which left in the funnel. The final produced biodiesel is then washed multiple times using hot water. The moisture and methanol avoided through rotary evaporator (AUTO ROTARY EVAPORATOR 30L, RE30V2) set at 62 °C. The yield of biodiesel was determined through Eq. (2) [2].

$$Yield of Biodiesel = \frac{Weight of CM biodiesel (g)}{Weight of CM leave soil (g)} * 100$$
(2)

Experiments Design

Response surface methodology (RSM) can be defined as an approach used to perform an experiment design that gives mathematical model of response surface from different sets of experimental runs. Utilizing the formulated mathematical equations, the response fluctuations with respect to parameter variations can be evaluated. Response surface methodology can be utilized to optimize parameters, refining models, and determining the goal of interest. With this paper, we have studied optimizing of operation conditions which are the key factors for production of biodiesel using central composite designs with four different factors. Central composite design (CCD) is another design of experiment that is helpful to develop second-order model and applying statistics methodology to find the required response variables with respect to the effects of interactions among various processing parameters. To develop the optimized model, reaction time of 15 to 7 5 min, 3:1 to 30:1 methanol to oil ratio, the reaction temperature (55 to 70 °C), and CaO NPs catalyst (0.5 to 8 wt.%) were considered (Table 1).

Table 1Experimental set up andindependent variable levels for	Parameters	Unit	Coded factors	Facto	r levels	
trans-esterification				- 1	0	1
	Temperature	C^0	Т	55	67.5	70
	Methanol:oil	mole	Μ	3:1	16.5:1	30:1
	Catalyst quantity	wt.%	С	0.5	4.25	8
	Reaction time	min	Rt	15	45	75

Results and Discussion

Nano-catalyst (CaO NPs) Characterization

Figure 1 indicates the functional group of CaO NPs which is extracted from eggshell and were determined by employing FTIR (Bremen, Germany) spectra with bands ranging from 4000 to 450 cm⁻¹. The peak/vibration belongs to functional groups from hydroxyl, alkenes, carbonyl, and carboxylic acids. The band at 876 cm⁻¹ corresponds to carbonate group (CO_3^{2-}) and stretching in CaCO₃. The bands at 1476 cm⁻¹ and 876 cm⁻¹ CaO NPs corresponding to aliphatic group (C-H)

The bands at 1476 cm⁻¹ and 876 cm⁻¹ CaO NPs corresponding to aliphatic group (C-H) show the CaO NPs carbonization. The bands at 3653 cm⁻¹ and 3454 cm⁻¹ associated to hydroxyl (O–H) group and resulting from moisture of water on the CaO NPs surface [19]. CaO NPs X-ray diffraction (XRD) analysis was performed to investigate whether structure of the synthesized nanoparticles is exactly CaO NPs or not. Figure 2 shows the result of XRD of CaO NPs used in this study (Bremen, Germany). It is seen that the results agree with Joint Crystal Powder Diffraction Standard (JCPDS) card number 77–2376.



Fig. 1 FTIR spectra of CaO NPs



Fig. 2 XRD analysis of calcium oxide nanoparticles (CaO NPs)

Importantly, the main peak happened at $2\theta = 38.55$ and the high-pitched and narrower spectral size in the XRD shape showed great polycrystalline nature of CaO NPs [4].

Figure 3 shows the surface morphology and microstructure of CaO nanoparticles derived from eggshell and was determined with help of SEM (JCM-6000PLUS, BENCHTOP SEM, and JEOL, JAPAN). The CaO NPs group is found to be spongy, and numerous porous with few particles and large piles was observed. The surface morphology reveals that the formed particles were of small size and leads to high porous surface magnification with 10 µm.



Fig. 3 CaO NPs SEM image

Characteristics parameter	Current value (%)	FAO/WHO standards	Literature value
Oil density@25 °C, (kg/m ³)	914	870	870–930
Kinematic viscosity (mm ² /s)	33.65	39.7	45-222
Refractive index	1.473	1.463	1.458-1.487
Saponification value (mg KOH/g)	185.79	181.40	96.4–199
Iodine value (ml/g)	95.98	80-106	64.1-79.95
Acid value (mg KOH/g oil)	3.18	4	2.4-5.6
Free fatty acids (%FFA)	0.64	0.57-0.728	0.33-1.7
Peroxide value (g/100 g)	2.6		0.5-7.6
Totox value (meq/kg)	9.38		
p-Anisidine value (meq/kg)	4.56		

 Table 2 Physicochemical properties of Croton macrostachyus leaf oil

Croton macrostachyus Leaves Oil Characterization

The *Croton macrostachyus* leaves oil quality is characterized based on their physical properties and fatty acid composition to make sure its selectivity as potential feedstocks in producing biodiesel. The analyzed result of physicochemical properties and fatty acid composition of *Croton macrostachyus* leaves oil studied in this work are summarized in Tables 2 and 3. The analyzed fatty acid composition shows that *Croton macrostachyus* leaf oil contains high percentage of palmitic acid (C16:0), Cis-11 eicosenoic acid (C20:1), linoelaidic acid (C18;2), and stearic acid (C18:0) acids. This shows *CM* leaves oil has a good capacity of stability and higher combustion quality [6]. The total molecular weight of *Croton macrostachyus oil* has been obtained from the fatty acid composition shown in Table 3 and was determined as 997.13 g mol⁻¹by using Eq. (1) as discussed in the "Extraction and Properties of CM Leaves Oil" section.

S. no	Test parameters	Test method	Test results		
1	Fatty acid composition (expressed as % of total fatty acid, w/w)				
	C6:0 (caproic acid)	AOAC 996.01(GLC)	0.82%		
	C8:0 (caprylic acid)		0.67%		
	C10:0 (capric acid)		2.54%		
	C12:0 (lauric acid)		1.97%		
	C14:0 (myristic acid)		1.47%		
	C16:0 (palmitic acid)		38.91%		
	C18:0 (stearic acid)		11.97%		
	C18:1 (cis-9 oleic acid)		8.48%		
	C18:2 (linoelaidic acid)		12.24%		
	C20:0 (arachific acid)		1.32%		
	C20:1 (cis-11 eicosenoic acid)		18.62%		
	C24:0 (lignoceric acid)		0.98%		

Table 3 Fatty acid composition of Croton macrostachyus leaf oil from experimental results

Statistical Analysis of the Experimental Results

The experiments for nano-catalyzed trans-esterification were undertaken for 30 runs as shown in Table 4. In the performed experiments, the optimum yield of biodiesel 96.375% was achieved with the optimal conditions for the selected four parameters investigated as reaction time 25.95 min, temperature 63.325 °C, molar ratio of methanol to oil: 28.093:1 in mg/L, and catalyst concentration 3.001% wt.

The optimized experimental data is processed using Design-Expert software version of 11.1.2.0 to generate the governing mathematical model for the prediction of ester content as a function of reaction time, reaction temperature, catalyst concentration, and molar ratio of methanol to oil. The obtained result shows that the full quadratic model of response

Run	A: Tempera- ture (°C)	B: Time (min)	C: Molar ratio (mole)	D: Catalyst(wt%)	Experimental yield (%)	Predicted yield (%)
1	50	15	30	0.5	76.53	78.63
2	85	15	30	0.5	59.36	60.31
3	50	75	3	0.5	65.04	86.56
4	67.5	45	16.5	4.25	88.01	89.37
5	67.5	45	16.5	11.75	85.44	87.2
6	67.5	45	16.5	4.25	95.20	95.22
7	50	75	3	8	81.04	82.7
8	50	15	30	8	68.89	62.45
9	67.5	45	43.5	4.25	93.46	94.94
10	85	75	30	0.5	82.94	86.46
11	50	75	30	8	77.51	72.41
12	32.5	45	16.5	4.25	38.30	35.36
13	67.5	45	16.5	4.25	94.9	95.47
14	50	15	3	8	94.13	66.21
15	67.5	105	16.5	4.25	58.67	91.48
16	67.5	45	16.5	-3.25	91.86	92.41
17	85	15	3	8	91.84	93.25
18	102.5	45	16.5	4.25	45.40	42.63
19	85	75	3	0.5	80.1	81.86
20	67.5	45	16.5	4.25	93.80	93.19
21	67.5	45	-10.5	4.25	91.20	96.29
22	67.5	-15	16.5	4.25	91.49	92.61
23	50	15	3	0.5	71.77	71.62
24	85	75	30	8	45.77	49.45
25	67.5	45	16.5	4.25	86.48	90.21
26	85	15	3	0.5	81.43	81.48
27	85	15	30	8	93.33	82.58
28	67.5	45	16.5	4.25	86.60	87.36
29	50	75	30	0.5	83.49	83.21
30	85	75	3	8	58.79	61.38

Table 4 CCD experimental design undertaken with four independent variables

Table 5 Coefficients of regression for biodiesel yield (%)	Predictors	Full quadratic		
prediction		Coefficient	p value	
	Constant	- 178.352		
	Temperature	6.8818	0.33	
	Methanol to oil ratio	0.6866	0.175	
	Reaction time	1.3957	0.6701	
	Catalyst	1.269	< 0.0001	
	Temperature* methanol to oil ratio	-0.0036	0.486	
	Temperature* reaction time	-0.0177	< 0.0001	
	Temperature* catalyst	-0.00209	< 0.0001	
	Methanol to oil ratio* reaction time	-0.136	< 0.0001	
	Methanol to oil ratio* catalyst	-0.0019	0.0002	
	Reaction time *catalyst	-0.223	< 0.0001	
	Temperature ²	-0.0009	< 0.0001	
	(Methanol to oil ratio) ²	0.044	0.185	
	(Reaction time) ²	-0.026	0.0017	
	Catalyst ²	0.224	0.0109	

surface is generated as the governing model for biodiesel production and applied for regression analysis as shown in Eq. (3). This equation can be applied to make predictions for the response value. Table 5 indicates the regression coefficients and corresponding p values. Variables in Eq. (3), "A" refers to temperature, "B" refers to reaction time, "C" refers to methanol to oil molar ration, and "D" refers to catalyst concentration. P values less than 0.0500 indicate model terms are significant. In this case, D, AB, BD, CD, and A² are significant model terms.

$$%FME = 91.80.9383A - 0.4067B + 0.53C - 6.88D - 9.34AB - 0.8612AC - 0.1375AD - 0.4612BC - 4.35BD - 5.54CD - 13.52A2 - 0.2550B2 + 63.75C2 + 1.06D2 (3)$$

Table 6 indicates that the developed quadratic model with regression coefficients of predicted ($R^2 = 0.8270$) is in reasonable agreement with adjusted ($R^2_{adj} = 0.9331$) showing better model for biodiesel production estimation; the difference is less than 0.2. Adequacy precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this work, the ratio of 22.803 indicates an adequate signal.

Figure 4 indicates the accuracy between the predicted model and actual model in scattered plot fully close to strait line which demonstrates a good agreement between the two results (actual and predicted). Reaction time, operating temperature,

Std. dev	4.59	R^2	0.9654
Mean	82.14	Adjusted R^2	0.9331
C.V. %	5.58	Predicted R^2	0.8270
		Adeq precision	22.8030

Table 6 Fit statistics



Fig. 4 Relation between predicted and actual yield (%) of a selected model





concentration of catalyst, and molar ratio are the important factors in the trans-esterification process that must be considered in the optimization of biodiesel synthesis. Since time is the primary parameter in the production of biodiesel, it must be minimized. Figure 5 illustrates the reaction time vs molar ration effect on the biodiesel yield. By keeping others factors constant and varying the reaction time from 15 to 30 min, it is observed that the increase of reaction time some variation enhances the yield biodiesel and reaches high yield of 93.375% at 22.95 min. Similarly, catalyst concentration has a significant role on the biodiesel production. Experiments were conducted by setting amount of catalyst to vary from 0.5 to 8wt%, keeping other parameters constant. It is observed that high yield of the biodiesel (96.375%) is obtained at 3.001 wt.%. Arumugam and Sankaranarayanan [4] obtained 96.3% at a catalyst amount of



temperature on yield at 57.75 °C reaction temperature and 16.5 wt.

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Fig.8 Response surface biodiesel content (yield) temperature vs. catalyst at 45-min reaction time and 16.5:1 methanol to oil ratio

4.25 wt% using residual ash from sugarcane leaf. Bahadur et al. [6] obtained 84.65% of biodiesel from *Croton gratissimus* oil utilizing sulfated zirconia oxide. Dubale et al. [11] studied the physical and chemical characteristic of *Croton macrostachyus leaves* and its components. Therefore, when compared to the reported work, our study gives better yield with a reasonable catalyst concentration. Figures 5, 6, and 7 indicate the 3-D surface plot which shows the combined effects of molar ratio, reaction time, catalyst, and temperature on biodiesel yield. Figures 8 and 9 indicate the interaction effects of temperature vs. catalyst and molar ratio of methanol to oil vs. temperature.

Validation of the Model

To validate the formulated model, three runs were conducted. As shown in Table 6, the model was validated experimentally at investigated optimal conditions (reaction time 25.95 min, temperature 63.325 °C, molar ratio of methanol to oil: 28.093:1 in mg/L, and catalyst concentration 3.001 wt%). The yield of biodiesel obtained from experiment (95.818%) approaches to the result determined from the developed model (96.375%) as shown in Table 7.



Fig.9 Response surface biodiesel content (yield) against methanol to oil ratio vs. temperature at 4.25 wt. (%) catalyst and 45-min reaction time

Physiochemical Properties of Produced Biodiesel

The *CM* oil biodiesel physiochemical properties were in the limit of ASTM standards as shown in the Table 8. It can be suggested that CM leaves can be applied as potential feed-stocks for producing biodiesel.

Table 7 Experimental data of model validation	Run number	Experimental biodiesel yield (%)	Predicted biodiesel yield (%)
	1	95.751	96.375
	2	96.251	96.375
	3	95.452	96.375
	Average	≈95.818	96.375

Properties	ASTM D 6751	CM oil biodiesel
Density @15 °C (g/cc)	0.82–0.9	0.84
Kinematic viscosity (mm ² /s)@40 °C	1.9-6.0	2.80
Cetane number (-)	>47 min	75
Flash point (°C)	>130 min	165
Water content (v/v)	0.05%max	0.02
Sulfated ash (w/w)	0.02%max	0.002
Acid value (mg KOH/g)	0.5max	0.2
Copper strip corrosion (-)	No.3max	2

Table 8 Physicochemical properties of CM biodiesel compared with ASTM standard

Conclusions

With this study, a response surface method (RSM) based on central composite design (CCD) has been utilized to formulate a viable experimental model to optimize biodiesel production from *CM* leaves oil. The effects of parameters including temperature, reaction time, amounts of catalyst, and methanol to oil molar ratio were studied. It can be concluded that the production of biodiesel from *CM* strictly affected by all these four factors. The developed optimal conditions which are used in producing biodiesel from *Croton macrostachyus* leaf oil amounts of CaO NPs, temperature, time, catalyst, and methanol to oil molar ratio were 63.325 °C, 25.955 min, 3.001 wt%, and 28.093:1, respectively. With these optimized conditions, the maximum attainable biodiesel percentage was observed approximately 96.375%. Therefore, it can be concluded that *CM* leaves oils is a high potential for the biodiesel production.

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Author contribution All authors contributed to research design and experimental work. The research work was conceptualized by Dr. Edo B. Jiru and Mr. Ermias G. Aklilu, and material preparation, data collection, and analyses were performed by Dr. Edo B. Jiru and Ramachandran Kasirajan, and supervision was performed by prof. Venkata Ramayya. The first draft of the manuscript was written by Dr. Edo B. Jiru, and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Data Availability All data analyzed during this study are included in this research article.

Declarations

Ethical Approval On behalf of all the co-authors, I am submitting the enclosed manuscript for potential publication only in Applied Biochemistry and Biotechnology. I attest that this paper has not been published in whole elsewhere and is prepared following the instructions to authors. All authors have contributed to this manuscript, reviewed and approved the current form of the manuscript to be submitted.

Consent to Participate Not applicable.

Consent to Publish All authors have read and agreed to publish the current version of the manuscript.

Competing of Interest The authors declare no competing interests.

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