Development of the Ethyl Ester from Jatropa Oil through Response Surface Methodology Approach

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ABSTRACT: With an increase in the global pollution, there is requirement for an alternative to the fossil fuels. Non-edible vegetable oils are highly promising for producing liquid fuels like diesel. Jatropha is a potential feedstock for biodiesel, currently utilized in India and many parts of the world. The optimization of reaction conditions such as temperature, time, catalyst and molar ratio for biodiesel production is important in reactor design. However, oils have characteristics reaction properties for optimum yield. Therefore, there is the need to identify such parameters in Jatropha oil ethyl esters production. Preparation of biodiesel from Jatropha oil ethyl esters using conventional homogeneous process. Optimization of Jatropha ethyl esters using Response surface methodology is done and data so obtained are fed to the design experiment software for analysis. The Jatropha ethyl esters yield was 92.62%. Maximum production of Jatropha oil ethyl ester was achieved with the process parameters viz molar ratio 8.5, reaction time 89.67min, reaction temperature 70.1°C and catalyst.0.62wt%.

Keywords: Jatropha oil; biodiesel production; reaction conditions; biodiesel oil.

INTRODUCTION

Energy has a vital part in the progress of a nation and is measured in terms of Economic and social development of that country (Chauhan et al., 2010; Chauhan et al., 2012; Chauhan et al., 2016). Measure of prosperity of a country depends on per capita energy consumption besides per capita income and GDP. Fossil fuels (petroleum oil, coal, and natural gas), renewable energy (hydro, wind, solar, geothermal, marine energy, and combustible wastes), and nuclear energy are

the major source of world energy consumption (Ansari et al., 2018; Sharma et al., 2019; Singh et al., 2018). Petroleum is the largest pool of energy consumption by the world. The ever-rising demand of transportation fuel has been fulfilled by Petro based fuels (approx. ~ 95%) (Cho & Chauhan, 2009). Processed vegetable oils (biodiesel) both edible & nonedible are being considered as a promising alternative to diesel for its use in a diesel engine (Chauhan et al., 2010, Chauhan et al., 2016, Cho & Chauhan, 2009; Chauhan et al. 2009; Rai et al., 2013; Singh et al., 2017). The impact of

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large-scale production of biodiesel and their usage are perceptible and a paradigm shift in consumption pattern has been visualized, and this is clearly encouraging the demand for biofuels (Singh et al., 2017; Singh et al., 2019).

The very fact that fossil petroleum diesel is limited and its long-term availability is required and the concern about the environmental norms and increasing pollution level forced to search the renewable alternatives in order to rectify current problems (Acharya et al., 2017; Agarwal et al., 2008; Agarwal et al., 2006; Algayyim et al., 2018). In recent decades, a balanced approach has been made by the scientists and researchers to increase the use of non- edible vegetable oil and biodiesel to draw a common policy nation wise to implement as genuine substitute to fossil diesel in engines without much modification the existing hardware (Ganesan et al., 2018; Sakthivel et al., 2013). Fuels from bio origins are also the solution of energy security, employment, restricting the country wealth used in importing fossil fuels, increasing agriculture economy, using waste land to grow oil rich crops and importantly environment pollution and degradation (Gautam & Kumar 2015; Gautam & Kumar 2018). A 10% biodiesel blend shows the best engine performance in terms of engine torque, engine power, fuel consumption and brake thermal efficiency among the all blending ratios for the three biodiesel blends (Ong et al., 2014). Blending J50C50 biodiesel with diesel reduces the carbon dioxide emissions and smoke opacity, but the nitrogen oxide and carbon increases monoxide emissions (Dharma et al., 2017). Literature review also confirms that methyl ester of Jatropha oil (JOME) has been extensively studied as a fuel with various approaches but ethyl ester of Jatropha oil (JOEE) as a fuel need to study because both the ingredients are renewable and can be easily derived from biomass (Singh et al., 2012).

Optimization of the yield of Papaya oil methyl ester was investigated methodology. using response surface Within the range of the selected operating conditions, the optimized values of temperature, catalyst amount, time, and methanol to oil molar ratio were found to be 62.33 °C, 0.95 wt %, 3.30 min, and 9.50:1 respectively. Based on the optimum condition, the predicted biodiesel yield was 99.9% and the actual experimental value was 99.3%. Papaya oil methyl ester (POME) exhibits property close to ASTM standards (Navak et al., 2019). The optimum combinations for the reaction were CALB:RML ratio (2.5:1), t-butanol to oil $(39.9 \square wt\%)$, temperature $(35.6 \square °C)$, methanol:oil ratio (5.9), reaction time $33.5\Box$ h. FAME yield of 78.3.5%, which was very close to the predicted value of 75.2%, was obtained (Shahedi et al., 2019). In the present study the different mechanism of biodiesel production is carried out with two stage integrated acidcatalyzed and pre-esterification of FFA and base-catalysed transeterification process to find the best techniques for production of fine biodiesel.

MATERIAL AND METHODS

Through response surface methodology (RSM) mathematical and statistical techniques are taken for analyzing problems by the influence of several independent variables and dependent variable or response, and the goal is to optimize this response (Tacias-Pascacio et al., 2019).

For response surface methodology, the dependent variable was viewed for the surface to which a mathematical representation was fitted. The regression equations were developed, related to various quality characteristics of biodiesel production, the second order response surface was assumed, as given in eq. (1):

$$Y = b_0 + \sum_{i=1}^{k} b_i x_i + \sum_{i=1}^{k} b_{ii} x_i^2 + \sum_{i=1}^{2} b_{ij} x_i x_j \pm e_r$$
(1)

The surface Y contains linear, squared and cross product terms of variables x_i s. It has been proposed that the scheme based on central composite rotatable design fits the second-order response surfaces quite accurately (Chauhan et al., 2016, Ansari et al., 2018, Sharma et al. 2018).

In this design, the standard error remains the same at all the points which are middle from the center of the region. This criterion of rotatability could be explained as follows: Let the point (0, 0, ---, 0) symbolize the center of the region in which the relation between Y and X is under investigation. From the results of any experiment, the standard error, er of Y can be computed at any point on the fitted surface. This standard error acts as a meaning of the co-ordinates x_i s of the selected point (Atapour et al. 2014; Awad et al., 2017; Hirkude & Padalkar, 2014). Thus, because of rotatability form, the standard error remains same at all equidistant points with the distance ρ from center of region i.e. for all points, this is given by the equation (2).

$$X_1^2 + X_2^2 + \dots + X_k^2 = \rho^2 = \text{constant}$$
 (2)

Central composite rotatable design is divided in three parts as-

- 1. Points related to 2^k design, where 2 is the number of levels where the parameters are kept during testing and k is the number of parameters
- 2. Extra points (star points), positioned on the co-ordinate's axes for a central composite design with star arm of size α
- 3. Additional points added at the center for equal precision to response Y of circle with radius one

Factor α gives the radius of circle or sphere on which the star points lie. For k \geq 5, experimental size is reduced for half replication of 2^k factorial design. Thus, α become 2^{(k-1)/4} for half replication. Also, no duplication is needed to find error mean square by replicating the center points (Bharawaz et al., 2016; Daud et al., 2018; Gopal et al 2018). In Table 1, components of second order middle composite rotatable design for a different number of variables are given.

Variables (k)	Factorial Point (2 ^k)	Star Point (2k)	Center Points (n)	Total (N)	Value of a
3	8	6	6	20	1.682
4	8	8	5	21	1.672
5	16	10	6	32	2.000
6	32	12	9	53	2.378

Table 1. Components of central composite second order rotatable design

The regression equation demonstrating second order response surface given by Eq. 3:

$$Y = b_0 + \sum_{i=1}^{k} b_i x_i + \sum_{i=1}^{k} b_{ii} x_i^2 + \sum_{i(3)$$

where, Y = estimated response, b" s are the coefficients and x_i " s are the independent variables.

As intended, both stages of transesterification i.e. acid catalyzed and base catalyzed would be optimized. The independent variables were chosen from the

preliminary studies conducted earlier which identified the most important factors affecting the esterification/ transesterification reactions of the Jatropha vegetable oils. For the esterification stage of methyl and ethyl both, concentration of catalyst (Para-toluene sulfonic acid-PTSA in this case), molar ratio, catalytic concentration, reaction. Time and reaction. Temperature were. Considered as the critical process, parameters, whereas for the alkaline stage, concentration of potassium hydroxide (KOH), reaction time and reaction. Temperature were considered. The design variables and levels are presented in table 2 for the esterification stage of Jatropha ethyl esters.

Table 2. Process Parameters and Their Levels For Esterification Jatropha Ethyl Esters

Coded Real	Deverations	1		Levels			
Factors	factors	Parameters	(-1.682)	(-1)	(0)	(+1)	(+1.682)
X_1	А	Molar Ratio	0.6	3.5	7.75	12	14.89
X_2	В	Catalyst Concentration (.% wt.)	.0.16	.0.5	.1	.1.5	.1.84
X_3	С	Reaction temperature (°C)	28.06	40	57.5	75	86.93
X_4	D	Reaction time (minute)	9.54	30	60	90	110.45

RESULTS AND DISCUSSIONS

The data so obtained were fed to the design experiment software for further analysis. The analysis of variance (ANOVA) and the statistical values were obtained from the software as shown in table 3. and table 4. The optimal FFA was .4.74% from the experimental design study. However, studies show that it should be less than.2% for higher yield. Therefore, esterification was done using the optimal parameters to obtain.4.74% FFA in.50 minutes. After that extra .20 minutes agitation given to get the requisite amount of FFA (<.2%). Moreover, the oil was transesterified as per the optimal process parameters of .0.88% of KOH by original oil mass and approx. 60°C temperature for.70 minutes. The sample from the reactor was gravity separated for.12 hours to remove glycerol. For purification, the sample was washed many times with lukewarm distilled water to remove traces of methanol/ethanol or catalysts that remained. After, the oil heated at.100°C for.30 minutes to evaporate the moisture contents. Lastly, Jatropha ethyl esters obtained as a light yellow and see through fluid. The. reaction. time, reaction. Temperature, molar. ratio and concentration of catalyst poly toluene sulphonic acid (.PTSA) are considered as the factors and the. % FFA as the response in the esterification stage.

Same procedure was followed as like esterification of methyl ester whereas the representation's F-value of.7.57 implied the representation is important. Probability of occurrence of such a F-value because of noise is very low about 0.14%. The terms in the representation would be important if

"Prob.> F" is less than.0.0500. In this case, B, .C, .AC, .AD, .BD, .C² are important representation's terms. For values more than 1000, the representation terms are not important. "Lack of Fit F-value" of .2.73 implied that the Lack of F is of no significance, relative to pure error. There is 17.47% probability that this huge "Lack of Fit F-value" could be present due to noise. It is known that a non-important lack of fit is desirable, so it is concluded that the representation fitted the design. ."Pred R-Squared" of 0.4153 is not very close to "Adj R-Squared" of 0.7472 as expected; i.e. the difference was greater than.0.2. This indicates a large-block effect or probably a problem with your representation and/or data. Things like a representation's reduction, response transformation, outliners are to be taken into account. "Adequate Precision" measured the signal to noise ratio. The required ratio was a value more than 4. So, a ratio of 10.475 indicated an adequate signal. These representations can help to steer through the design space.

Equation of coded factors make predictions about response for given levels of each factor with +1 for high levels of the factors and -1 for lower levels. Relative impact of factors can be calculated by comparing factor coefficients with the help of coded equation.

The prediction of response for the levels of each factor can be done by applying the equation using actual factors. Here, levels have been specified in the original units for.each factor. This equation could not.be used to determine relative impact of each factor, because the coefficients are multiplied in order to keep the units of each factor in check and the intercept is obtained away from the center of the design space. As, all representation's statistics and diagnostic plotted are important, the Representation's Graphs icons are finished up. Statistical considerations are satisfied for1) Normal probability plot to find the normality of standardized residuals.

2) Standardized residuals versus estimated values: Check for constant error.

3) To monitor influential values for externally standardized residuals

4) For power transformations using boxcox plot.

ANOVA. for Response Surface Reduced Quadratic representation's									
	Analysis. of variance [Partial sum of squares]								
Source	Sum of Squares	df	Mean Square	F-Value	p-value Prob> F	Remarks			
Representation's	52.60	9	5.84	7.57	0.0014	Significant			
A-Molar ratio	2.00	1	2.00	2.59	0.1359	-			
B-Catalyst concentration	11.52	1	11.52	14.92	0.0026				
C-Reaction temperature	4.56	1	4.56	5.91	0.0333				
D-Reaction time	1.41	1	1.41	1.83	0.2035				
AC	7.03	1	7.03	9.10	0.0117				
AD	8.64	1	8.64	11.19	0.0065				
BD	9.10	1	9.10	11.78	0.0056				
C^2	16.72	1	16.72	21.66	0.0007				
D^2	1.680E-003	1	1.680E-003	2.176E-003	0.9636				
Residual	8.50	11	0.77						
Lack of Fit	7.02	7	1.00	2.73	0.1747	Non-Significan			
Pure Error	1.47	4	0.37			C			
Total	61.10	20							

Table 4. Statistical values of the Ethyl Esterification of Jatropha oil

Std. Dev.	0.88	R-Squared	0.8610
Mean.	4.58	Adj R-Squared	0.7472
C.V. %	19.20	Pred R-Squared	0.4153
PRESS.	35.73	Adeq Precision	10.475
-2 Log Likelihood.	40.59	BIC	71.03
-		AICc	82.59



Fig. 1. Estimated and Actual Values for Esterification Jatropha Ethyl Ester

Fig.1 shows the estimated and the actual values of the design. It may be clearly seen that the estimated and actual values grouped are quite similar and concentrating on a medium range. In the light of the above discussions, it may be stated that the representations had effectively estimated the %FFA for large sized esterification with minimal error. It was actual that the estimated and the actual %FFA are very close in all the cases validating the previous assumption that the representation was statistically important. Now the three-dimensional surfaces were plotted using the representation in design experiment software. Response surface graphs represent the main and interactive effects of the factors on the response (% FFA). Results so obtained are shown in Fig. 2, Fig. 3 and Fig. 4, respectively.

Relationships between the parameters are indicated by the two independent variables that are plotted and converted on three-dimensional contour lines. In Figure 2, the change in the conversion of FFA having a molar ratio of ethanol.to acidified oil with reaction temperature are shown. It is seen that at high reaction temperature, increase in molar ratio leads to increase in conversion of FFA and at high molar ratio decrease in reaction temperature led to decrease in FFA conversion. If biodiesel is produced using a solid catalyst, reaction kinetics show that the reaction depends on the temperature at one stage and the results are in line with similar type of work (Anwar et al., 2018; Elango et al., 2019).



Fig. 2. Response Plot for Effect of Reaction Temperature and Molar Ratio on Esterification of Ethyl Ester



Fig. 3. Response Plot for Effect of Reaction Time and Molar Ratio on Esterification of Ethyl Ester

Figure.3 shows that the change of FFA attains a peak value.at low molar ratios of ethanol to acidified oil and longer reaction time. Increasing the molar ratio of ethanol to acidified oil does not improve the conversion of FFA due to decreased concentration of active catalyst sites in the reaction (Dharma et al., 2016, Atapour et al., 2014). Also, hydrogen bonds between ethanol and sulfonic group (-SO₃H) of the catalyst form easily due to the polarity of ethanol and ethanol molecules adsorbed on active sites of the catalyst. Concentration of ethanol reached a certain level, then due to the poor accessibility of catalyst surface, esterification was stopped. Therefore, high conversions were obtained at long reaction time and low molar ratios of ethanol to acidified oil.

Fig. 4 presents the response plot for reaction time and catalyst loading with respect to the conversion of FFA. Thus, long reaction time and high amount of catalyst aided high conversion of FFA. Also, with reduced catalyst action and reaction times, conversion of FFA decreased. This occurs as the percentage of catalyst loading reduces which decreases the number of reactants adsorbed on the active sites of the catalyst. And the conversion of FFA increases with increase in the reaction time (Esonye et al., 2019; Karmakar et al., 2018). Optimum FFA of 1.9 % was given by the representation and catalysts concentration of 1.0%, with reaction temperature equal to 59 °C and reaction time of 72 minutes. Thus, the catalyst concentration reduced the FFA value below 2%, which is recommended for transesterification. However, looking at the initial FFA of 13.7 % the reduction was found to be encouraging.

The table 5 shows the analysis of variance for the transesterification of ethyl ester. The Representation's F-value of 592.87 implied the representation held importance. An F-value being large due to noise was unlikely, with just 0.01% chance. Values of Prob> F, less than 0.0500 shows that representation's terms are important. For such case, A^2 , B^2 , C^2 , D^2 , B, C, D, BC, CD, AD shows important representation's terms. For values more than 0.1000 shows that representation's terms are not important. Lack of Fit F-value as 1.18 prove that Lack of Fit was not important relative to pure error. 44.89% of Lack of Fit F-value is due to noise. Lack of fit as non-important is desired (Elango et al., 2019; Dharma et al., 2016; Esonye et al., 2019). The statistical values for Transesterification are as shown by Table 6.



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Fig. 4. Response Plot for Variation of Esterification of Ethyl Ester with Reaction Time and Catalyst Concentration

ANOVA for Response Surface Reduced Quadratic representation							
Analysis of Variance [Partial sum of squares]							
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob> F	Result	
Representation	2295.53	11	208.68	592.87	< 0.0001	Significant	
A-Molar ratio	0.067	1	0.067	0.19	0.6727	-	
B-Catalyst Concentration	25.21	1	25.21	71.61	< 0.0001		
C-Temperature	1539.15	1	1539.15	4372.70	< 0.0001		
D-Time	15.38	1	15.38	43.69	< 0.0001		
AD	36.00	1	36.00	102.26	< 0.0001		
BC	19.59	1	19.59	55.67	< 0.0001		
CD	17.64	1	17.64	50.12	< 0.0001		
A^2	11.82	1	11.82	33.59	0.0003		
\mathbf{B}^2	15.02	1	15.02	42.68	0.0001		
C^2	659.24	1	659.24	1872.87	< 0.0001		
D^2	7.37	1	7.37	20.93	0.0013		
Residual	3.17	9	0.35				
Absence of Fit	1.89	5	0.38	1.18	0.4489	Non-Significant	
Absolute Error	1.28	4	0.32			c	
Corr. Total	2298.70	20					

Table 5. Analysis of V	Variance for	Transesterification of E	thyl Ester

Table 6. Statistical Values for Transesterification of Ethyl Ester

Std. Dev.	0.59	R-Squared	0.9986
Mean	85.49	Adj R-Squared	0.9969
C.V. %	0.69	Pred R-Squared	0.9859
PRESS	32.40	Adeq Precision	86.411
-2 Log Likelihood	19.88	BIC	56.41
-		AICc	82.88

Pred R-Squared of 0.9859 results in Adj R-Squared of 0.9969; i.e. the difference was less than 0.2. Signal to noise ratio was measured by Adeq Precision. Ratio more than 4 was required. Ratio of 86.411 gives desirable signal. This representation can also be used to steer through the design space.

Ethyl Ester = $+91.50-0.070^{*} \text{ A}+2.11^{*}$ B+10.62* C+1.06* D+3.30* AD+1.56* BC-1.48* CD-0.89* A²-1.00* B²-6.64* (4) C²-0.70* D²

Equation 4 of coded factors make predictions about response for given levels of each factor with +1 for high levels of the factors and -1 for lower levels. Relative impact of factors can be calculated by comparing factor coefficients with the help of coded equation.

Ethyl Ester = -27.94301 - 1.57967 * A + 24.94843 * B + 2.99756 * C + 0.16572 * D + 0.031020 * A * D + 0.44714* B * C - 3.39429E -003 * C * D - 0.049245* A²-25.06558* B²-0.021688* C²-1.12336E-003* D² (5)

Equation 5 for actual factors is for making assumptions about the response for different levels of each factor.

When statistical conditions are fulfilled, the results are shown in figure 5.

Here, levels have been specified in the original units for each factor. This equation could not.be used to determine relative impact of each factor, because the coefficients are multiplied in order to keep the units of each factor in check and the intercept is obtained away from the center of the design space. When statistical conditions are fulfilled, the results are shown in Figure 5.



Fig. 5. Estimated and actual values Transesterification Jatropha Ethyl Ester



Fig. 6. Response Plot for Variation of Transesterification of Ethyl Ester with Molar Ratio and Time

Figure 6 is response surface plot of the effect of molar ratio and time with constant catalyst concentration and temperature and the influence of reaction time and quantity of ethanol on the JOEE yield. Molar ratio (ethanol/oil) is most important for the

transesterification for producing biodiesel and the separation, recovery of glycerol from oil depends on it. 6:1 is considered the best molar ratio for methanol, in the preset study molar ratio of 9:1 is best for ethanol. The biodiesel yield increased at higher time and high ethanol/oil ratio because the conversion of triglycerides increases with time and excess ethanol favored the complete conversion to biodiesel (Sharma et al., 2019, Ansari et al., 2018, Singh Y., 2015).



Fig. 7. Response Plot for Variation of Transesterification of Ethyl Ester with Catalyst Concentration and Temperature



Fig. 8. Response Plot for Effect of Time and Temperature on Transesterification of Ethyl Ester

Figure 7 presents the response surface plot of the variation in biodiesel yield with temperature and catalyst concentration when the ethanol-oil ratio is 7.75 during 75 minutes. NaOH acts as catalyst at elevated temperatures as transesterification reaction is faster than saponification reaction. Thus, temperature is main factor to increase the rate of reaction for producing biodiesel. At lower molar ratio (3:1) yields increases with increase in catalyst concentration. At higher molar ratio (7.5:1), ethyl ester produced, increased. Hence, in improving the ethyl ester yields, catalyst concentration is the main variable. Biodiesel produced has problems in separation if too much catalyst is used, due to emulsion formation. Too much catalyst, over 1.0wt % decreased biodiesel production due to soap formation that inhibits ester layer formations (Sharma et al., 2019, Jamshaid et al., 2019, Yatish et al., 2018).

Figure 8 presents the response surface plot of the variation in biodiesel yield with

time and catalyst concentration when the ethanol-oil ratio was 7.75:1 at 0.8% wt. as catalyst concentration obtains the yield 93.5%. Biodiesel yield increased with higher catalyst concentration. The usage of too much catalyst led to saponification reaction and reduced biodiesel yield (Sharma et al., 2019, Atapour et al., 2014, Sathish Kumar et al., 2015). Optimum of estimated 92.62 % was by the representation's and catalysts concentration of 0.62%, molar ratio 8.5: 1, reaction temperature of 70.12°C and reaction time of 89.67 minutes for minimize the glycerol and maximize the production of ethyl ester. Also, ethanol can be used as a substitute for the methanol in optimized conditions. Thus, biodiesel can be made from nonconventional sources (like ethanol obtained from sugar cane, soybean oil). This study shows the usage of ethanol or methanol along with Jatropha oil to produce fuel.

CONCLUSIONS

The present study was done on Jatropha oil which was converted into Jatropha ethyl ester and optimization for production of Jatropha oil ethyl esters using response surface method. RSM proved to be a powerful tool for the optimization of biodiesel production. It has been reported that the yield of biodiesel was high enough to produce biodiesel in a practical way.

- Maximum production of Jatropha oil ethyl ester was achieved with the process parameters viz molar ratio 8.5, reaction time 89.67min, reaction temperature 70.1°C and catalyst.0.62wt%. An empirical relationship has been developed to predict the product yield incorporating transesterification process parameters at 92.62 %. \pm 0.3 %.
- Confidence level for the studies on different mechanism of biodiesel production revealed that two stage integrated acid- catalyzed preesterification of FFA and base-

catalyzed transeterification process may be considered the best for production of biodiesel.

- RSM proved to be a powerful tool for the optimization of biodiesel production. It has been reported that the yield of biodiesel was high enough to produce biodiesel in a practical way. The optimal conditions for the maximum methyl ester yield were found to be at catalyst concentration of 0.57% w/w, methanol/oil molar ratio of 7.16, and a reaction temperature of 59.43°C.
- An empirical relationship has been developed to predict the product yield incorporating transesterification process parameters at 95% confidence level. The predicted value for yield is found 94.48%. The error in the experimental and the statistical model results was only 2.08%

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CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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