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Optimization of long term storage conditions (temperature, period and antioxidant concentration) of waste frying oil biodiesel by RSM.

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Abstract: Biodiesel getting its reorganization as a fuel, alternative to fossil fuel whose coffers are being exhausted rapidly. Thus, the fuel gets an acceptance as the future source because of its sustainability, renewability and environment friendly in nature. In the present work, the storage conditions of waste frying oil biodiesel was optimized by using a statistical approach - Response Surface Methodology. The antioxidant Pyrogallolconcentration (150, 300, and 450 ppm), storage temperature (15, 30, and 45°C) and storage period (1.5, 3, and 4.5 months) were taken as the input variables. Based on the Box-Behnken experimental design, 15 experimental runs were carried by varying the input conditions at different levels. Acid values and Viscosity were considered as the output responses. The increase in viscosity and acid values were observed from samples stored at dissimilar storage conditions. However, the acid value did not surpass the biodiesel standard limit set by EN 14214. The viscosity values of biodiesel were observed to increase above the maximum limit of 5.0 cSt. The optimum storage condition to maintain the viscosity within the limit of 5.0 cSt was determined and a model equation was developed to determine viscosity in terms of storage temperature, antioxidant concentration and storage period using the response optimizer technique. The developed model equation predicted the viscosity with a correlation coefficient of R^2 value equal to 0.9926. The storage conditions of biodiesel were optimized with 435 ppm of antioxidant concentration, 45°C as storage temperature, and 4 months storage period which is also verified by genetic algorithm.

Keywords: Response surface method (Box Behnken Design), Waste frying oil, Optimization, Storageconditions, Viscosity.

1. Introduction

Limited usage and vacillation in the price of existing conventional fossil fuel directed the research toward the renewable fuels as substitute. Biodiesel is a potential stand-in for diesel oil. Biodiesel (100%) or its blends with diesel are used directly for many applications in many countries. It is ensued from different sources like crop or non-crop oils, waste fats, lards, etc., thereby adjusting the need of petroleum imports. The advantages of biodiesel over diesel fuel are; it is a sustainable low sulphur fuel. It is biodegradable. It has comparable fuel properties like heat content, ignition quality, viscosity, and volatility with that of diesel. Its higher flash point ensures it safer to transport and stock up. Biodiesel has around 10 – 11% oxygen content which helps in further combustion completely ensuing in lower HC, smoke, and CO gas emissions [1].

Biodiesel's prime drawbacks is its stability at some period in storage. Storage stability of biodiesel is its resistance to change from its initial composition and properties when stored in a given

environment for a certain period time. The biodiesel's stability is lower compared to diesel fuel and consequently, the combination of biodiesel to diesel fuel reduces the stability of the biodiesel - diesel blend [2]. The instability of biodiesel fuel is mostly due to its unsaturated FAME content. The unsaturated fatty acids easily deteriorate and form undesirable products. Biodiesel stored in open containers comes in contact with atmospheric air and undergo auto-oxidation reactions. Biodiesel on exposure to heat suffers thermo oxidative reaction. Whereas, in contact with moisture in air or storage tanks undergo hydrolysis reaction. Such reactions produce undesirable components such as alcohols, aldehydes, peroxides, and hydro- peroxides in biodiesel and thereby increases the acidity, change the physio-chemical characteristics of biodiesel, and deteriorates its fuel quality [3-6]. Certain products may undergo polymerization reactions and produces gums and insoluble sediments with higher molecular weight. The fuel properties that are mainly affected by the degradation reactions during storage are density, viscosity, flash point, acid number, iodine, and peroxide values. The use of low- quality biodiesel in the engine may cause fuel filter plugging, injector fouling, carbon deposits, corrosion of fuel system components, decrease in thermal efficiency, and reduction in engine life[7,8]. Storage stability may possibly be affected by contact with lights, contaminants, sediments, change in colour reduces the fuel clearness [9,10]. Hence, there is considerable concern on maintaining the quality and stability while storing biodiesel among biodiesel manufacturers, suppliers, and consumers.

Many works on the study of biodiesel's storage stability obtained from both edible / inedible oils were carried out. And also few studies on the effect of physical properties of biodiesel quality on its long-term storage and explored the outcome of the fuel properties pertaining to time and biodiesel quality during its long- term storage [10 –20]. The premier work on the testing of storage stability was carried by De Villiers, Du Plessis, and Vander Walt in 1985. The influence of storage conditions such as contact to daylight, ambient air,antioxidants, , metal impurities and temperature on storage stability were studied and reported. The authors investigated the biodiesel's storage stability derived from sunflower oil at a different temperature, the influence of antioxidant (TBHQ), influence of contact with steel, andexposure to light. The storage stability was measured in terms of viscosity, induction period, freefatty acid content, peroxide value, and anisidine value [21].

Mittelbach and Gangl investigated the changes in physio-chemical characteristics of biodiesel obtained from rapeseed oil and waste frying oil stored for 170 - 200 days at a temperature of 20-22°C under open/close to air, exposed /covered to sunlight and contact to metal (stored in tin cans). During storage under light and oxygen, the viscosity increased due to the polymeric compounds formation [22]. Bondioli et al., analyzed the storage stability of biodiesel samples derived from rapeseed oil, sunflower oil, waste cooking oil, tallow, etc. The samples were stored in metal containers, open space, under direct sunlight that prevailedin summer and winter for one year. During the period of storage, the viscosity was seen to increase slightly and constantly for all the samples [23]. The effect of storage stability on long term from biodiesel prepared from vegetable oils like low and high erucic Brassica carinata oil, sunflower oil with high oleic content respectively, and waste cooking oil was studied by Bouaid et al., Results showed an raise in insoluble impurities, acid value, peroxide value, and viscosity and a reduce in iodine value [15]. Tang et al., investigated the effectiveness of a variety of synthetic and natural antioxidants such as BHT, BHA, IB, PG, TBHQ, DTBHQ, PY, and tocopherol on the biodiesel's stability produced from oil of cottonseed and soybean, yellow grease, and poultry fat. Antioxidants with concentration of 250-1000 ppm were used. It is observed that the stability could be improvedwith PG, PY [24].

Shahabuddin et al., evaluated the storage stabilities of palm biodiesel, coconut oil and jatropha derived biodiesels. The changes in fuel properties (acid value, density, flash point,

viscosity) were measured at an interval of 180 hrs throughout 2160 hrs. The results reveal an boost in viscosity, acid number and density due to oxidation. The content of unsaturated fatty acid in biodiesel has a better impact on the stability of the biodiesel [25]. Mazumdar et al., examined the significance of temperature and ambient conditions of storage stability on the Jatropha biodiesel for 12 months. The storage temperatures were 4°C, 25°C, and 35°C with environmental circumstances exposed to air, with and without light. The investigation showed an increase in viscosity, acid value and density for an increased time in storage [26]. Christensen and McCormick observed the stability storage based on experiments (accelerate oxidation by keeping samples at 43°C) for one year. Antioxidants were added before and after aging experiments. It was found that for retaining stability, it is required to treat aged biodiesel with antioxidants [27]. Fu et al., investigated the stability of storage of waste cooking oil biodiesel contained 60% of unsaturated fatty acid esters. The physio-chemical properties such as acid number, peroxide value, density, viscosity, phase behavior and the heat of combustion were monitored and observed that the viscosity, density, acid number and the peroxide values increased significantly during the storage period [28].

Several studies are available in the literature on the use of antioxidants to improve the biodiesel's storage stability. The antioxidants inhibit the oxidation reactions by eliminating free radicals produced by the initiation stage reaction of oxidation. Natural antioxidants like tocopherols, pyrogallol, beta-carotene, selenium, lutein, lycopene were commonly used. The stability improvement by effectiveness of natural antioxidants is found very small when compared to that of synthetic antioxidants [11, 29].

Fröhlich evaluated the effect of naturally occurring tocopherols (alpha, gamma, and delta), carotene, and astaxanthin on the biodiesel's stability. The antioxidants - biodiesel mixtures were exposed at 65°C in the presence of air. Gamma tocopherol was found most effective and the antioxidant's effectiveness was found to depend on biodiesel composition [30]. Shyamala et al., experimented to study the antioxidant capability of leafy vegetables such as spinach, cabbage, Hong one and coriander leaves on storage of heated (upto frying temperature) groundnut oil and sunflower oil stored for 1 month. The result showed that leafy vegetables are excellent antioxidants [31]. Mittelbach and Schober experimented the influence of various synthetic antioxidants (PY, PG, BHA, TBHQ and BHT) on the stability of biodiesel obtained from tallow, sunflower, rapeseed and waste frying oil. The antioxidants concentration used was from 100 - 1000 ppm. The significant improvement observed in induction period of biodiesel produced from waste frying oil, rapeseed, and tallow with TBHQ, PG and PY. It was concluded that the effectiveness depends on the antioxidants chemical structure [32].

Sendzikiene et al., compared the oxidative stability of biodiesel produced from fats of animals, rapeseed and linseed oils. It was found that biodiesel from vegetable oil are further stable than those obtained from animal fat. In addition, it is suggested that 400 ppm of synthetic antioxidants (BHA and BHT) are required to stabilize biodiesel [33]. Polavka et al., examined the OS of waste fried oil and fresh rapeseed oil derived biodiesels both treated by pyrogallol or BHT by using the Rancimat method and found that the biodiesel's stability increases noticeably with the pyrogallol as an antioxidants than that of BHT [34]. Domingos et al., investigated the effectiveness of BHT, BHA and TBHQ of soybean oil biodiesel stability. It was found that BHT to be the effective antioxidant with a concentration of 0.70 WT% and TBHQ produces an enhanced stabilizing at 0.80 WT% concentrations [35]. Xin et al., (2009) showed that safflower biodiesel with high content polyunsaturated fatty acids is less stable compared to palm biodiesel that contains high saturated fatty acid content. It was also shown that the PG is a high efficient stabilizer

compared to DPPD [36]. Araújo et al., (2009) analysed the performance of TBHQ, BHA and PG on stability of biodiesel from castor oil. The result exhibits that that BHA is better than PG and TBHQ [37].

The storage stability of Karanja oil derived biodiesel at dissimilar storage environment was srrudied by Das et al. The different storage conditions were experimented with and without contact with air, sunlight, exposure to the metal, with antioxidants etc., for 180 days. The effectof adding different antioxidants like PY, PG, TBHQ, BHA on the stability was also studied. Antioxidants were used in the concentration of 100, 200, 300, 400, 500 and 1000 ppm. The resultshowed an increase in viscosity and peroxide value with respect to storage period. The samples kept under exposure to atmosphere and dark, open to the elements of metal and exposed to outside air showed the highest improve in its viscosity and peroxide value compared to samples stored at some other conditions. Among all antioxidants, PG was found the most excellent followed byBHT and BHA [14].

Jain and Sharma showed that the TBHQ is the most beneficial antioxidant followed by PY. The natural antioxidants (including tocopherol and vitamin E) are ineffective compared to synthetic antioxidants [38]. Jain and Sharma analyzed the long term stability on storage of jatropha methyl ester. Samples of biodiesel using metal concentration 0 – 2 ppm and PY concentration 0-800 ppm were stored open to air, sunlight, and at ambient temperature. Jatropha biodiesel fuels without any metal contamination were found unstable. The PY concentration of 200 ppm added could maintain the stability of jatropha biodiesel for six months. Peroxide value, acid value and viscosity were begun to augment towards storage period without the adding up of antioxidants. But, increases in the above properties were regulated by the addition of additives PY [13]. Dantas et al. probed the OS of corn biodiesel during long-run storage and heating at 150°C. The result confirmed the enhance in viscosity, peroxide, and iodine value of corn biodiesel by the oxidative decomposition. The fuel degradation was found to be very high under heating conditions [39].

Lapuerta et al. scrutinized the controlled oxidation in biodiesel derived from waste cooking oil, soybean oil and animal fat with anti-oxidant addition using the Rancimat method. The result showed better stability for more saturated biodiesel fuels. Waste frying oil biodiesel was found unstable caused by the loss of natural anti- oxidants, formation of contaminants and foreign compounds during the frying process [40]. Yang et al. analyzed the consequences of the storage period against the weathering processes of biodiesel obtained from canola oil, animal fat, and oil from soybean under open air and ambient temperature for 190 days. The result found that the evaporation rate of FAME is worse than diesel because of its high boiling point of the former [41]. Yang et al. explored the effect of solvents (methanol, water, and acetone), metals Cu, Pb, Al steel), fatty acid composition, and antioxidants (α-tocopherol, PY, PG, BHT, BHA, TBHQ) affecting the stability of canola oil, soybean oil, and tallow biodiesels. The samples were stored in a sealed steel container and maintained at − 4°C with no light. The result reveals that the acid numbers were not depending on the fatty acid structure. The addition of PY or TBHQ showed significant improvement in the induction period [42].

Serrano et al. made a survey on the stability of three commercial antioxidants (synthetic and natural) for oxidation stability of rapeseed, soybean, high oleic sunflower oil and palm derived biodiesel. Synthetic additives were hindered phenol, hindered phenol/amine, PG, and natural antioxidant mixed tocopherols. The result indicated that more effective for improving the biodiesel stability by PG and tocopherol- based antioxidants [43]. Fattah et al. reviewed that the phenolic antioxidants are greater effectual than amine antioxidants. Further, it was reported that PY is efficient to enhance the OS of biodiesel derived from various feedstock such as vegetable oils, waste frying oil, tallow's, grease and fats [44]. Sulistyo et al. analyzed the use of four antioxidants

(synthetic) PY, PG, TBHQ, and BHT on jatropha biodiesel (23.5%w/w saturated). The biodiesel was found to have low oxidative stabilityof 1.37 h of the induction period. Antioxidants were added on a concentration of 100 – 2000 ppm. The PY with 204 ppm showed the best improvement in stability. The results indicates the effectiveness order with PY followed by PG, BHT and TBHQ [45].

Sarin et al. distinguished the influence of alpha-tocopherol (α-T) (natural antioxidant) and BHT, TBP, OBPA, and TBHQ (synthetic antioxidants) on OS of jatropha curcas biodiesel by its induction period (IP) by Rancimat test at 110 ˚C. The result shows that synthetic additives are much beneficial than natural even at low concentration for restoring the stability of biodiesel from jatropha [29]. Jain and Sharma investigated the effect of antioxidant (PY) and metal impurities on the long term storage stability of biodiesel derived from jatropha. Correlations were developed between induction period and antioxidant concentration, storage time, and metal concentration using response surface methodology. For jatropha biodiesel contaminated with Cu,Co, Mn, and Ni it was found that the storage time is 2.07, 2.76, 3.24, and 3.62 months respectively with the above metal and antioxidants concentration [46]. Pavalavana Pandian and Pugazhvadivu studied the influence of storage stability of waste cooking oil and its diesel blend by considering the factors like storage period, storage temperature, and antioxidant concentration. To maintain the acid value and viscosity at standard, a correlation was developed and observed that pyrogallol as an antioxidant shows a good result [47, 48].

Dwivedi et al. optimizedthe storage conditions using RSM for a 6 months of storage period of Karanja biodiesel in the presence of metal contaminants (Fe, Al, and Zn) , antioxidant (PY). Three process variables storage duration, metal, and antioxidant concentration were taken as a process variables and the induction period was measured as a response. Mathematical model equations were developed to determine the IP. The addition of metal contaminants decreased stability while PY improved was observed. The Addition of PY also helped to store the jatropha biodiesel in these metal containers [49].

Fatima Shafiq et al obtained the highest yield of 90.6% biodiesel from chicken fat oil by the various reaction parameters optimization viz the catalyst concentration, time of reaction and temperature and ratio of methanol-to-oil mole based on the RSM [50]. Sara Maen Asaad et al used the RSM to enhance the performance of biodiesel in engines by optimizing the process variables of biodiesel applications. The optimized combination of values, input process parameters, experimental design used, output response parameters and the competence of the model and response for the studies were discussed [51]. Taiebeh Tamorad et al determined the optimum setting for the production of biodiesel through RSM based on Box–Behnken design including the observation of 3D contour plots and calibration curves [52]. Jehad Yamin et al studied on the Modeling the effect of catalyst concentration, oil to methanol ratio and reaction time on the waste cooking oil biodiesel using RSM [53].

It is observed from the above studies that biodiesel are more vulnerable to oxidation and gets deteriorated during storage. The various factors that influence the storage stability are found to be physiochemical properties, lack of antioxidant, storage temperature, and duration of storage. In literature, waste frying oil generated from a single source is generally considered for the study. Further storage stability of biodiesel, various storage conditions were not taken for study simultaneously. In the this work, the waste fried oils were collected from sources that use different types of cooking oils like groundnut, palm, sunflower oil etc., The storage condition such as antioxidant concentration, storage temperature, and storage time of waste frying oil biodiesel is

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optimized based on RSM technique

2. Methods

The biodiesel involved in this study was prepared from 5 litres of Waste frying oil sample procured from a snack shop and its fatty acid composition was measured using GC-FID (Chemito - GC 8610). The catalyst NaOH, methanol, and Pyrogallol (antioxidant) are purchased from the local chemical supplier of Sigma Aldrich, India. The optimization of storage conditions for the long-term storage stability of waste frying oil biodiesel was carried out by Response surface methodology (Box-Behnken optimization technique). The experimental run in coded variables based on Box Behnken design was obtained using MINITAB 17.0.1 software. The storage conditions like the concentration of antioxidant (A), storage temperature (T), and storage period (P) were considered as input parameters for optimization. The cold temperature of the samples were maintained by a refrigerator (Make: Cryo scientific CSLR 422 of temperature range -8°C to 20°C) and the hot temperature is maintained by a thermostatically controlled food warmer (Make: FW554 of temperature range 30°C to 80°C). Table 1. Shows the selected input parameters along with their coded and uncoded levels as required by the Box Behnken design matrix. The optimized values were compared with the Genetic Algorithm (GA) using MATLAB.The sample was converted into biodiesel by one step transesterification process and this biodiesel was used for the storage stability analysis. The properties such as density, viscosity and acid value of biodiesel were determined.

Table 1 Coded and uncoded levels of Box-Behnken design

Input Parameters	**Symbols**	**Coded and uncoded level**		
		-1	**0**	**1**
Antioxidant concentration(ppm)	A	150	300	450
Storage temperature (°C)	T	15	30	45
Storage periods (in months)	P	1. 5	3. 0	4. 5

Biodiesel Preparation

Biodiesel was prepared by taking methanol to waste frying oil in the molar ratio 6:1 and 1 wt% of NaOH. Methoxide solution was prepared by mix in 10 g of NaOH pellets in 200 ml of methanol. NaOH is preferred in the transesterification process because of its lower water content, gives a higher yield of biodiesel compared to KOH or other catalysts. One litre of waste frying oil was taken in a round bottom flask and preheated to 55°C. Then, the solution of methoxide was mixed slowly with the waste frying oil sample. The resulting mixture was heated for one hour with constant speed of stirring at 250 rpm. Then the heated combination was transferred into a separating glass funnel which is kept undisturbed until two layers were seen. The top biodiesel layer was separated from the layer of lower glycerin. Distilled water is used to wash biodiesel for three times to eliminate the traces of catalyst and glycerol.

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Sample Preparation

The samples for the experiment were prepared according to the design of experiment produced by Box Behnken technique. Table 2. shows the coded variables of 15 experimental runs. The experiments were conducted according to the combination of input parameters. In this work, pyrogallol was used as an antioxidant. The concentration of antioxidant 150, 300, and 450 ppm were measured by using electronic weighing scale .The samples to be maintained at 15° were kept in the refrigerator used to preserve medicine and other samples that are to be controlled at 30°C and 45°C were kept in a food warmer. All the 15 samples were filled in a 250 ml with airtight lid glass bottle. The experimental run in coded variables based on Box Behnken design was obtained using MINITAB 17.0.1 software (Table 2). For example, for the experimental run 1, 150 ppm of pyrogallol was added to biodiesel and it was maintained at 15°C of temperature and stored for a 3 months period. At the end of each experiment, the acid value and viscosity of the samples were measured.

Table 2 Design of Experiment by Box Behnken Design

Experimental Run	Antioxidant Concentration (A) in ppm	Storage Temperature (T) in °C	Storage period (P) in months
1	-1 (150)	-1 (15)	0 (3.0)
2	1 (450	-1 (15)	0 (3.0)
3	-1 (150)	1 (45)	0 (3.0)
4	1 (450)	1 (45)	0 (3.0)
5	-1 (150)	0 (30)	-1 (1.5)
6	1 (450)	0 (30)	-1 (1.5)
7	-1 (150)	0 (30)	1 (4.5)
8	1(450)	0(30)	1(4.5)
9	0(300)	-1(15)	-1(1.5)
10	0(300)	1(45)	-1(1.5)
11	0(300)	-1(15)	1(4.5)

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12	0(300)	1(45)	1(4.5)
13	0(300)	0(30)	0(3.0)
14	0(300)	0(30)	0(3.0)
15	0(300)	0(30)	0(3.0)

Experimental Design

The technique Response surface methodology (RSM) has been used to investigate the influence of storage conditions like storage period, antioxidant concentration, and storage temperature on the biodiesel's long- term storage stability in terms of Acid value and Viscosity. A three-factor, one replicate Box Behnken design (BBD) including three center point proceeding to 15 runs, was used for response surface fitting. The storage conditions like the concentration of antioxidant (A) in ppm, storage temperature (T) in °C, and storage period (P) in months were considered as input parameters for optimization. The optimized values were compared with the Genetic Algorithm (GA) using MATLAB. The Genetic algorithm is a method of solving constraint and unconstrained optimized value based on natural selection. Using the experimental results, the output results namely viscosity and acid value were fitted with the quadratic model given in Eqn. 1 [54].

$$Y = \beta 0 + \sum_{i=1}^{k} \beta ixi + \sum_{i=1}^{k} \beta i1xi^2 + \sum i < \sum j \beta ijXiXj + \varepsilon \quad (1)$$

where Y = response parameter (acid value and viscosity).
X = Input parameters (A, T, P).
β_k = regression coefficient.
ε = term of error with a normal distribution with standard deviation of σ and mean of 0.

3. Results and Discussion

The waste frying oil sample collected from the snack shop is analyzed with gas chromatography. The fatty acid composition of biodiesel from waste frying oil that was used for storage stability analysis is given in Table 3. The oleic acid, linoleic acid and palmitic acid were ound to be the major fatty acids. The total saturated, unsaturated, and DU are calculated as 41.78%, 56.73%, and 69.72% respectively.

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Table 3 Composition of Fatty acids in waste frying oil

Name of the Fatty acids	Composition (%wt)
Myristic acid (C14:0)	0.98
Palmitic acid (C16:0)	35.11
Stearic acid (C18:0)	4.91
Oleic acid (C18:1)	43.98
Linoleic acid (C18:2)	12.51
Linolenic acid (C18:3)	0.24
Arachidic acid (C20:0)	0.35
Behenic acid (C22:0)	0.11
Lignoceric acid (C24:0)	0.32
Other acids	1.48
Total saturated fatty acid (%wt)	41.78
Total unsaturated fatty acid (%wt)	56.73
Degree of unsaturation (DU)	69.72

The properties of waste frying oil biodiesel that is used for long-term storage stability analysis is presented in Table 4. Properties like viscosity, density, acid value, cetane number and iodine value are found to be within the limits specified by EN14214 standards.

Table 4 Properties of biodiesel used in long-term storage stability analysis

Properties	Biodiesel
Density(g/cm^3)	0.867
Viscosity@40°C(cSt)	3.681
Heating value (MJ/kg)	39.85
Calculated Cetane number	56.66
Flashpoint(°C)	204
Calculated iodine value (g I_2 /100 g of oil)	71.31
Acid value (mg KOH / g of oil)	0.206

Table 5 shows the measured acid and viscosity values of the biodiesel samples that are stored according to different storage conditions mentioned in the Box-Behnken design matrix. From Table 5, it is seen that an increase in the acid value of biodiesel that is stored at different conditions. However, the acid values did not exceed the limit (EN 14214 specification: max 0.5 mg KOH / g of oil). Hence, the acid value was not considered as the response parameter in the optimization study. From Table 5, it is further seen that the increased viscosity ofbiodiesel from its initial value after storing under dissimilar storage conditions (The viscosity of biodiesel before storage is 3.681 cSt). It is also seen that at certain storage conditions, the viscosity of biodiesel has crossed its maximum limit of 5.0 cSt. For example, biodiesel stored with an antioxidant concentration of 300 ppm for a storage period of 4.4 months at 15°C shows the highest increase in viscosity of about 66.50%.

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Table 5 Measured acid and viscosity values of biodiesel samples at differentstorage conditions

Experimental runs	Input parameter (Coded)			Input parameter (Uncoded)			Output parameter	
	A	T	P	A	T	P	Acid value (mgKOH/ g ofoil	Viscosity (cSt)
1	-1	-1	0	150	15	3	0.2413	6.514
2	1	-1	0	450	15	3	0.2336	8.977
3	-1	1	0	150	45	3	0.2965	6.354
4	1	1	0	450	45	3	0.3003	4.521
5	-1	0	-1	150	30	1.5	0.2995	4.554
6	1	0	-1	450	30	1.5	0.2612	5.244
7	-1	0	1	150	30	4.5	0.3105	5.987
8	1	0	1	450	30	4.5	0.3195	5.879
9	0	-1	-1	300	15	1.5	0.2826	5.421
10	0	1	-1	300	45	1.5	0.2642	7.366
11	0	-1	1	300	15	4.5	0.3112	10.987
12	0	1	1	300	45	4.5	0.3212	5.266
13	0	0	0	300	30	3	0.2486	4.912
14	0	0	0	300	30	3	0.2482	4.911
15	0	0	0	30	30	3	0.2479	4.912

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Fig 1 shows the values of the viscosity of biodiesel for different experimental runs. The viscosity of biodiesel stored at five different storage conditions (experimental run 4, 5, 13, 14, and 15) are found within the limit of 5.0 cSt. For the remaining 10 storage conditions, the viscosity exceeded 5.0 cSt. The experimental run 11 has the highest viscosity value of 10.987 cSt. The increase in viscosity may be due to the oxidative degradation reactions that occured at these storage conditions [23, 32, 38].

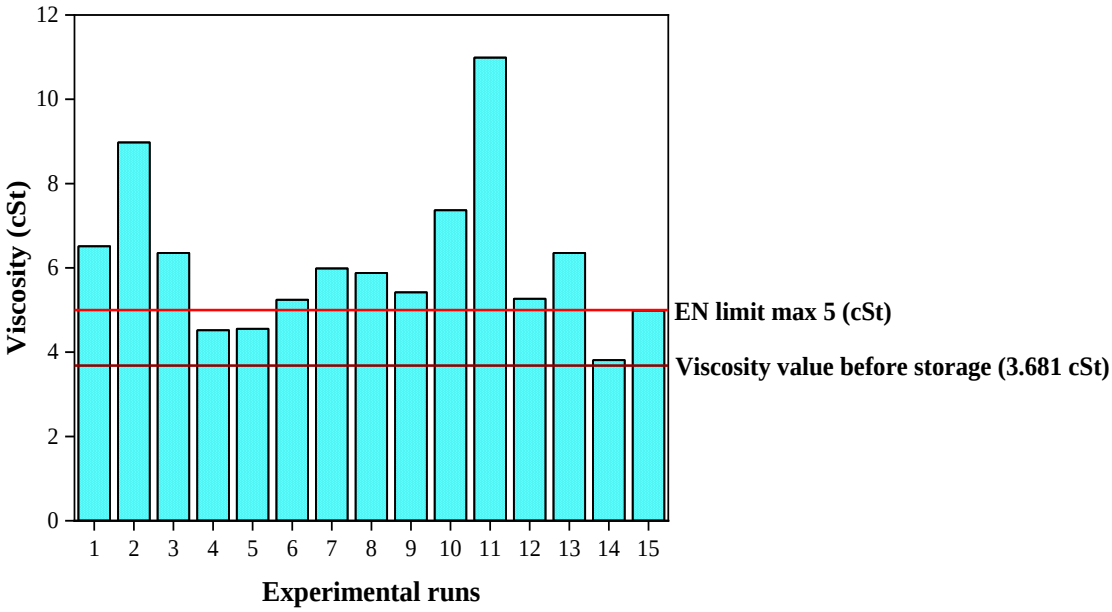

Fig 1. Viscosity value of biodiesel before and after storage

By using response surface methodology, the experimentally determined values of viscosity are statistically fitted and a mathematical model was formed to predict viscosity based on Box-Behnken design using MINITAB 17.0.1. Using the coefficients obtained from the predicted model for temperature, storage period and antioxidant of the biodiesel in terms of uncoded factors for viscosity is specified by the Eqn (2)

$$\text{Viscosity} = 4.912 + (0.1515 \times A) - (1.0490 \times T) + (0.6918 \times P) - (0.082 \times A^2) + (1.762 \times T^2) + (0.586 \times P^2) - (1.074 \times A \times T) - (0.199 \times A \times P) - (1.917 \times T \times P) \quad (2)$$

Table 6 shows the predicted values and experimental values of viscosity. The mathematical model is given in equation (1) can predict the result accurately. The predicted values and experimental values of viscosity were analysed which shows a deviation of less than 6%of error. In addition, it was also with a regression coefficient value of 0.9926, which gives a less deviation from the predicted and original values. The adjusted R square value is found to be 0.9783 and the predicted R square as 0.8818. The scatter plot between experimental and predicted viscosity value of waste frying oil is drawn. The degree of fitness of the predicted model can also be visualized in Fig 2. The correlation coefficient R^2 is 0.9926, which shows a higher degree of fitness of the developed model equation that predicts viscosity.

Table 6 Experimental and predicted values of viscosity

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Experimental runs	Antioxidant (Pyrogallol) concentration (A)	Storage temperature (T)	Storage period (P)	Experimental viscosityvalue (cSt)	Predicted viscosity value(cSt)	Error (%)
1	-1(150)	-1(15)	0 (3.0)	6.514	6.415	1.51
2	1 (450)	-1(15)	0 (3.0)	8.977	8.866	1.24
3	-1(150)	1 (45)	0 (3.0)	6.354	6.465	-1.75
4	1 (450)	1 (45)	0 (3.0)	4.521	4.620	-2.19
5	-1(150)	0 (30)	-1(1.5)	4.554	4.373	3.97
6	1 (450)	0 (30)	-1(1.5)	5.244	5.074	3.24
7	-1(150)	0 (30)	1 (4.5)	5.987	6.155	-2.81
8	1 (450)	0 (30)	1 (4.5)	5.879	6.060	-3.08
9	0 (300)	-1(15)	-1(1.5)	5.421	5.699	-5.13
10	0 (300)	1 (45)	-1(1.5)	7.366	7.437	-0.96
11	0 (300)	-1(15)	1 (4.5)	10.987	10.918	0.63
12	0 (300)	1 (45)	1 (4.5)	5.266	4.984	5.36
13	0 (300)	0 (30)	0 (3.0)	4.912	4.912	0.00
14	0 (300)	0 (30)	0(3.0)	4.911	4.912	-0.02
15	0 (300)	0 (30)	0(3.0)	4.912	4.912	0.00

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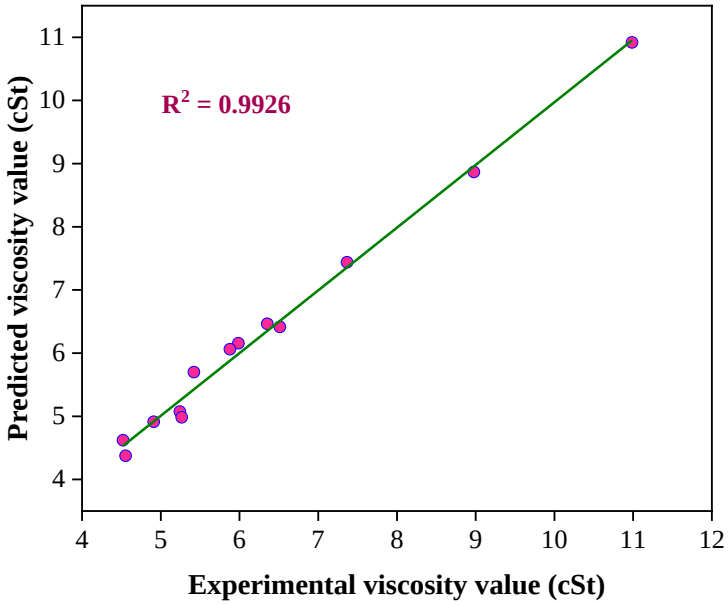

Fig 2. Scatter plot of measured viscosity vs. predicted viscosity values.

The adequacy and significance of the second-order developed model can be determined by ANOVA. Table 7 shows the result of ANOVA of the predicted model. The F- value obtained from the regression model is 74.67, which is greater than the F table value (4.77 at 95% significance). It confirms the adequacy of the model fit. Further, Table 7 shows that the linear, square, and interaction terms also have a significant effect on predicting the viscosity. Further, the standard error of noise S is 0.2579. A Lower value of S indicates a better fit. The R- sq (adj) and R-sq (pred) are found to be 0.9793 and 0.8818 respectively. The higher these values better are the fit.

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Table 7 Analysis of variance for viscosity prediction.

Source	DF	Adj SS	Adj MS	F	P
Regression	9	44.7303	4.9700	74.67	0.000
Linear	3	12.8150	4.2717	64.18	0.000
Square	3	3.8281	4.1501	62.35	0.000
Interaction	3	19.4650	6.4883	97.48	0.000
ResidualError	5	0.3398	0.0666	***	***
Lackof Fit	3	0.3328	0.1109	332788.50	0.000
PureError	2	0.0000	0.0000	***	***
Total	14	45.0631	***	***	***

The three- dimensional response surface plots were developed to evaluate the impact ofthe combination of the independent variable (biodiesel's storage conditions) on the viscosity of the biodiesel. The plot shown in Fig. 3 – 11 represents the relationship between two factors (input variables) and one response (output variable). Fig. 12 - 15 is the two-dimensional contour plots that shows the surface plot which connects identical responses in the same contour line as constant response. Fig 3, 4, and 5 (3D surface plot) indicates the dependency of viscosity of the biodiesel on storage temperature and antioxidant concentration. The lower viscosity is obtained at higher antioxidant concentrations and with lower storage temperature. Fig. 6, 7, and 8 (3D surface plot) indicates the dependency of the viscosity of the biodiesel on storage period and antioxidant concentration. Higher storage period with low antioxidant concentration gives lower the viscosity of the biodiesel. Similarly Fig. 9, 10, and 11 (3D surface plot) indicates the dependency of viscosity of the biodiesel on storage period and storage temperature. Higher the viscosity is obtained by maintaining higher storage period and antioxidant concentration. Fig. 12,13, and 14 (contour plots) indicates the dependency of viscosity on the input variables such as storage period, storage temperature, and antioxidant concentration by holding the code values as m-1, 0 and 1 (minimum, zero and maximum level of storage conditions).

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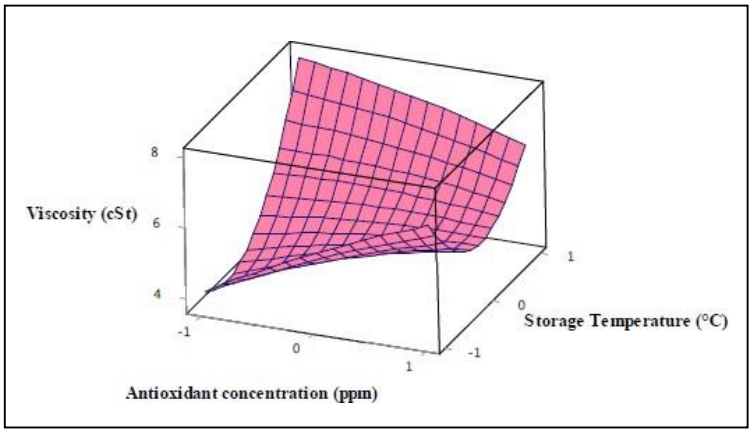

Fig. 3 Response surface curve (storage period at a minimum level)

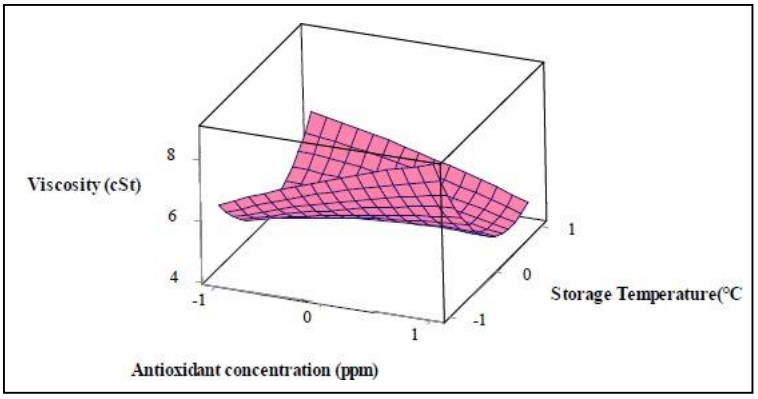

Fig. 4 Response surface curve (storage period as middle level)

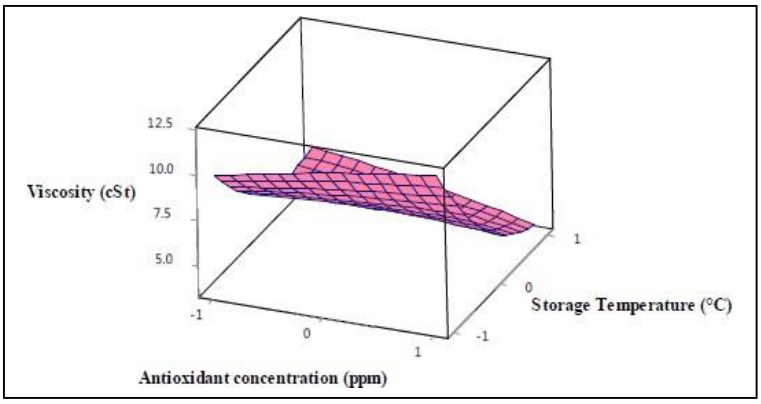

Fig. 5 Response surface curve (storage period at a maximum level)

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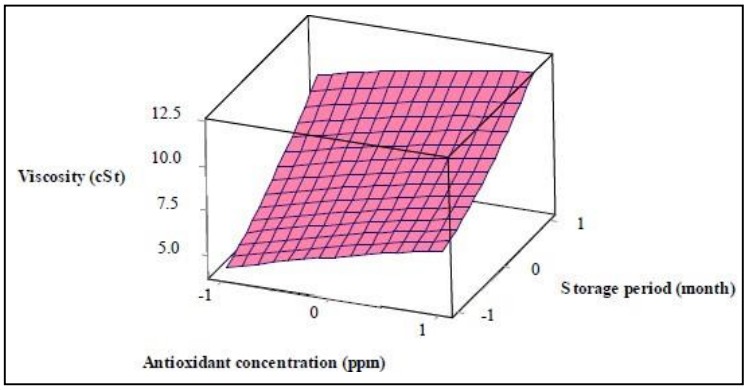

Fig. 6 Response surface curve (storage temperature at a minimum level)

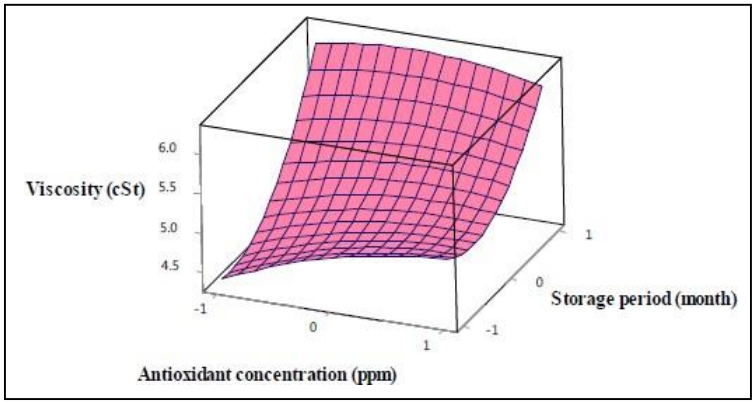

Fig. 7 Response surface curve (storage temperature at centre level)

.

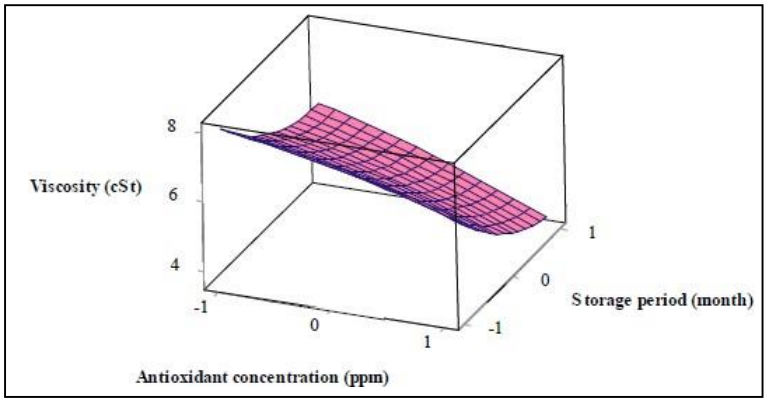

Fig. 8 Response surface curve (storage temperature at maximum level)

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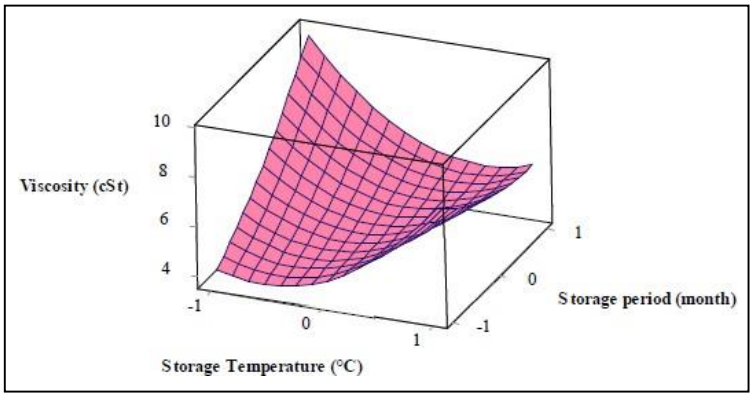

Fig. 9 Response surface curve (antioxidant concentration at minimum level)

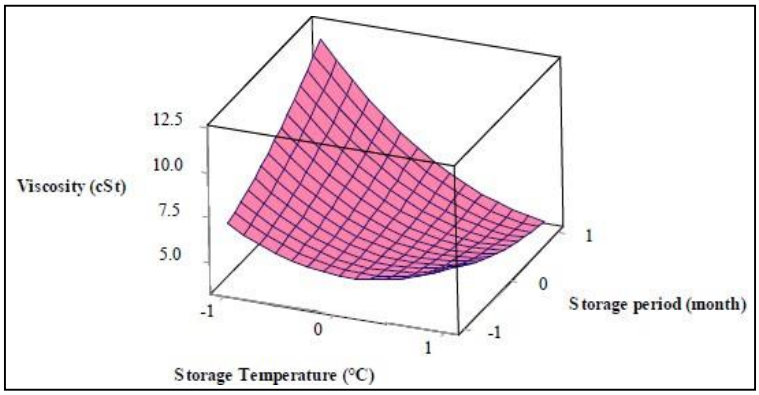

Fig. 10 Response surface curve (antioxidant concentration at centre level)

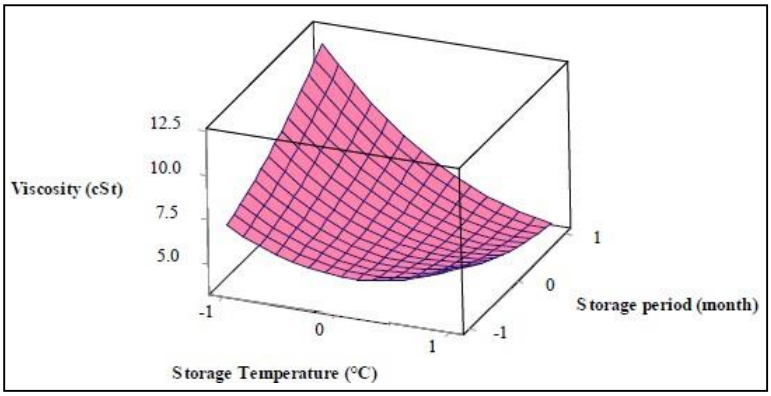

Fig. 11 Response surface curve (antioxidant concentration at maximum level)

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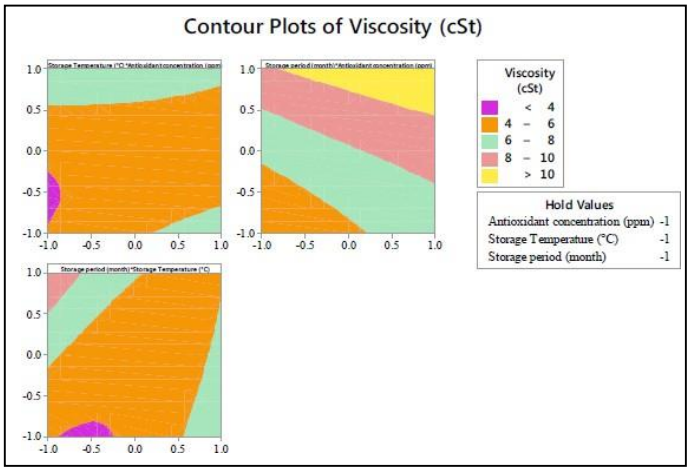

Figure 12 Contour plots of viscosity (parameters at minimum level)

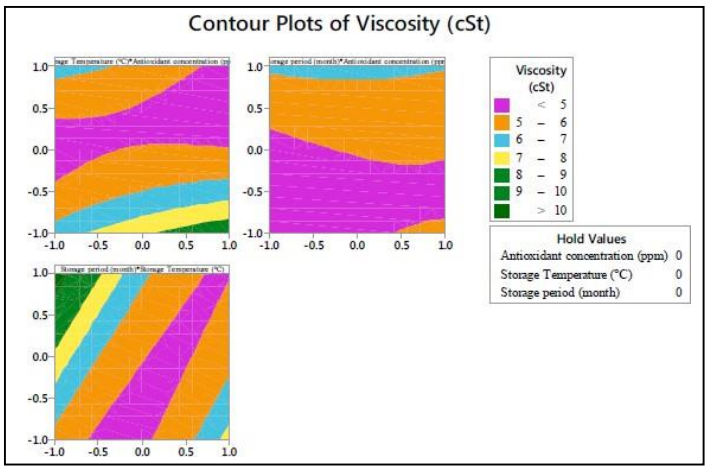

Figure 13 Contour plots of viscosity (parameters at middle level)

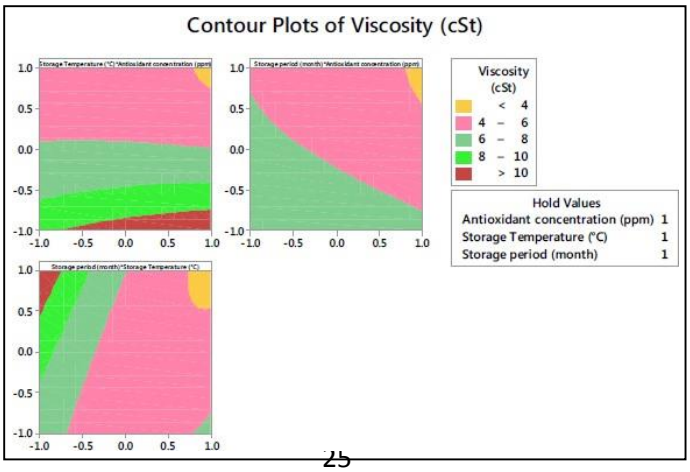

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Fig 14 Contour plots of viscosity (parameters at maximum level)

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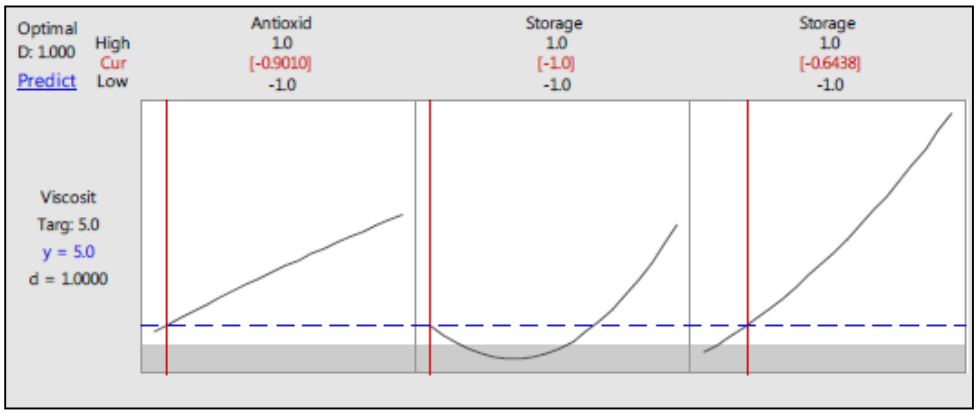
As the response surface curves and contour plots are complex, it is very much needed to identify the optimum input variables to maintain the viscosity of biodiesel within the 5 cSt as per the EN standard. The optimization work related to this is carried out using response optimizer in the MINITAB 17.0.1 and also using constraint optimization program developed based on Genetic Algorithm (GA) of MATLAB 13. The results of constraint optimum conditionsof input variables are obtained from MINITAB 17.0.1 response optimizer given in Fig 15.

Fig 15 Optimization plot from response optimizer

Similarly the constraint optimum condition of input variables using MATLAB 13. GA constraint optimization



programs are compared in Table 8. The error was calculated from theresults obtained by RSM and GA techniques.

Table 8 Viscosity predicted through RSM and GA

Experimental runs	Predictedviscosity value (cSt) RSM	Predictedviscosity value (cSt) GA	Error (%)

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1	6.415	6.409	0.094
2	8.866	8.859	0.079
3	6.465	6.459	0.093
4	4.620	4.617	0.065
5	4.373	4.367	0.137
6	5.074	5.069	0.099
7	6.155	6.151	0.065
8	6.060	6.059	0.017
9	5.699	5.691	0.140
10	7.437	7.431	0.081
11	10.918	10.911	0.064
12	4.984	4.98	0.080
13	4.912	4.909	0.061
14	4.912	4.908	0.081
15	4.912	4.909	0.061

Both the results indicate that optimum conditions of output variables are within the range. The input variables coefficients obtained from the optimizer plot is given below:

Antioxidant concentration (A) in coded value (ppm) = $\zeta_A = 0.9010$
Storage temperature (T) in coded value (°C) = $\zeta_T = 1$
Storage period (P) in coded value (months) = $\zeta_P = 0.6438$

The relationship between coded and actual values of input variables are given below

- Antioxidant concentration $A = 300 + 150\zeta_A$ (3)
- Storage temperature $T = 30 + 15\zeta_T$ (4)
- Storage period $= 3 + 1.5\zeta_P$ (5)

Substituting the coded values of ζ_A, ζ_T and ζ_P in Eqn. 3, Eqn. 4 and Eqn. 5 respectively, the actual values of optimized input variables are obtained as,

$A = 435.15$ ppm ≈ 435 ppm $T = 45°C$

$P = 3.96$ months ≈ 4 months

The optimized storage conditions for maintaining storage stability (measured in-terms of viscosity) of biodiesel derived from waste frying oil are antioxidant (Pyrogallol) with 435 ppm concentration 435 ppm, the storage temperature at 45°C and 4 months of storage period. The viscosity is maintained within 5 cSt , while the waste frying oil biodiesel is kept under the above storage conditions.

4. Conclusions

Biodiesel prepared from waste frying oil collected from snack shop and its fatty acid composition and its initial properties such as acid value, viscosity and density were experimentally determined. The experiments were designed according to Box-Behnken RSM at different conditions of temperature, time, and antioxidant concentrations to find the long-term storage stability of biodiesel. The properties of biodiesel samples stored at various conditions are examined after a storage period of 1.5, 3, and 4.5 months maintained at 15, 30, and 45°C with an antioxidant concentration of 150, 300, and 450 ppm as per the design of experiment generated by using Box Behnken Method by MINITAB 17.0.1 software. The properties were determined after each storage condition. The deviation in the properties with the initial was compared and analyzed. It was observed that the acid value of all samples were within the standards limit as per EN14214. It is further noted that the viscosity value gets changed and exceeds the standard limit during storage period. To maintain the viscosity at 5 cSt, a correlation is developed by using RSM – Box Behnken technique. The correlation produces the predicted viscosity very closer to the experimentally determined values. The accuracy of the equation is compared with the GA using MATLAB and found to be in good agreement. The R^2 value of the regression coefficient was found to be 0.9926. The optimized

storage condition was obtained as 435 ppm of antioxidant to be added with biodiesel maintained at 45°C for the period of 4 monthsholds the viscosity within the standard limit of 5 cSt.

List of Abbreviations

BHA - Butylated hydroxyanisole
BHT - Butylated hydroxytoluene
IB - Ibuprofen
PG - Pyrogallate
TBHQ - tert-Butylhydroquinone DTBHQ - Ditert-Butylhydroquinone
PY - Pyrogallol
TBP - Tributyl phosphate
OBPA - 10,10-Oxybisphenoxarsine
DPPD - N,N´-Diphenyl-p-phenylenediamine
OS - Oxidation stability
IP - Induction Period
RSM - Response Surface Methodology
DU - Degree of Unsaturation
Cu - Copper
Co - Cobalt
Mn - Manganese
Ni - Nickel
Pb - Lead
Al - Aluminium

Declarations:

Availability of data and materials

The datasets generated and/or analyzed during the current study are available from the corresponding author on reasonable request.

Competing interests

The authors declare that they have no competing interests.

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Authors' contributions

JPVP and SS contributed to the conceptualization and methodology, performed data analysis and simulation. Both reviewed and edited the manuscript. All authors read and approved the final manuscript

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.

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