Oxidation stability of Biodiesel: A review

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Abstract- The world is going out on crisis of conventional petroleum fuels either due to their unavailability or their harmful effect to health and environment. Biodiesel has emerged as a potential alternative to petroleum diesel due to its chemical characteristics similar to petroleum diesel with less harmful environmental effects. Biodiesel is mixture of fatty acids alkyl esters derived from triglycerides present in oils and fats. The problem with biodiesel is its oxidative action on storage. On oxidation biodiesel molecules are transformed into peroxides which further breaks into acids, alcohols and esters. The extent of biodiesel oxidation is determined like Iodine value, oxidisability, structure Indices etc. The oxidation stability is affected factors like structure, atmospheric content etc. In this study an attempt has been made to study the oxidation mechanism and characteristics of biodiesel, factors affecting stability of biodiesel and parameters used to determine the extent of oxidation of biodiesel.

Index Terms- Biodiesel, Oxidation stability, Oxidation Chemistry, Biodiesel Stability Parameters, Stability Factors

Index-

I. Introduction

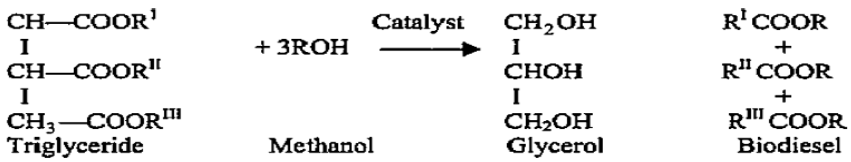
Biodiesel is the environmental friendly alternative to conventional petroleum diesel fuel, derived from the feedstock like oils (edible and non edible oils) and animal fat. Biodiesel consists of alkyl esters produced from the transesterification of the triglycerides in the presence of acidic or basic catalyst [1-3]. Fuel crisis and environmental degradation are the major concerns which the world is confronting now. Countries round the globe are dependent almost entirely on liquid petroleum fuels for public and private transportation. Due to rapid industrialization the demand of petroleum fuel increased manifold, the dwindling supply of petroleum reserves has become more and more concentrated in a few oil-producing countries and all other countries are largely dependent on these oil producing countries for the oil supply. Transportation, agriculture as well as industrial sectors are largely dependent on the fuel supply. Indiscriminate extraction and consumption of fossil fuels has already reduced the petroleum reserve significantly. This indiscriminate extraction and consumption results the rapid rise in prices of crude oil. The crude oil prices has shown the steep rise from 20$/barrel to about 140$/barrel and then back to nearby of 71$/barrel since 1999-2000 to 2018-2019[4,5].This growing dependency, increased import bills, uncertainty in availability and increasing environmental concern have encouraged many countries to adopt programs to search for alternative liquid-fuel produced from domestic and renewable resources. The desire of replacing petroleum fuels with a more reliable and sustainable energy source has created much interest in bio-fuels. In addition, Biofuels especially the biodiesel is gaining popularity in commercial use due to the adaptation of the biodiesel with the current transportation, agricultural and industrial infrastructure with no or little modification.

Countries like USA and Europe are using the biodiesel derived from edible oil and animal fat in engines in place of petroleum diesel. Contrary, Production of biodiesel from edible oil is not easily possible in India, as India imports about 60% of edible oil to meet the domestic requirements [6]. The possible source of biodiesel production in India is found to be the non edible oil resources like Jatropa curcas, pongamia etc. As per Government of India Survey, India has about 60 Mha as waste and degraded land i.e. not suitable for regular agriculture, 5 million Kms as road network and 67,000 kms as railway network. This waste land and sides of road and railway network can be used to cultivate jatropha curcas and biodiesel can be produced from the grown seeds [7-9]. This biodiesel can be proved to be the potential substitute for diesel demand. Similar to vegetable oils and animal fat, the presence of unsaturated components leads to the susceptibility of biodiesel to the oxidative degradation. This susceptibility to oxidative degradation is helpful in rendering the biodiesel biodegradable in nature. With storage, biodiesel becomes chemically unstable and quality of the fuel is degraded. This oxidation process creates the problem of injector chocking, fuel line and fuel filter chocking and depositing of the sediments in combustion chamber and fuel supply lines.

Various other factors that affect the stability of the biodiesel are presence of light, air, heat, antioxidants, impurities, material of the container used for storage. Along with these the stability of biodiesel is also affected by the method use for the purification of biodiesel during the manufacturing process. In this study an attempt has been made to study and review the oxidation stability of biodiesel due to storage.

II. PROCESS OF BIODIESEL PRODUCTION

Vegetable oils can be chemically modified to produce biodiesel through micro-emulsion, pyrolysis (thermal cracking) or transesterification. Transesterification is the most common method used for biodiesel production and yields the best quality biodiesel having higher cetane number, lower exhaust emission and better combustion properties than biodiesel produced from any other method. This transesterification reaction is carried out by continuous intermixing of vegetable oil (triglycerides) with alcohol in presence of catalyst under suitable temperature range [10].



The overall transesterfication reaction is carried out in three reversible reaction. The first is the conversion of triglycerides into diglycerides, followed by conversion of diglycerides into monoglycerides and then monoglycerides into glycerol along with the formation of ester molecule in each step [11].

The transesterification process of biodiesel formation is affected by several factors such as molar ratio of oil to alcohol, temperature of reaction, catalyst type (acidic or basic), catalyst concentration, reaction time, free fatty acid (FFA) and water content of the feedstock used [12].

III. BIODIESEL STABILITY AND CAUSE OF INSTABILITY

According to Westbrook, “stability of biodiesel is its ability to resist the physical and chemical changes caused by interaction with the environment”. The poor stability is due to the presence of the double bond in free fatty acids.

Fatty acids on the basis of carbon bond present are as follows:

A. Saturated fatty acids, no double is present in between two carbon atoms.
B. Unsaturated fatty acids, carbon- carbon double bonds are present.

Unsaturated fatty acids can be further classified as:

A. Monounsaturated fatty acids (MUFA), contains one C-C bond.
B. Polyunsaturated fatty acids (PUFA), containing more than one C-C bond separated by a single methylene group. Example Linoleic and Linolenic acid.

The factors affecting the oxidation stability of fatty acids are:

A. Polyunsaturation
B. Position of C-C bond
C. Number of allylic and bis-allylic sites [12].

Bis-allylic carbon sites are more susceptible to oxidation attack as compared to allylic sites in fatty acids. Allylic position equivalent (APE) and bis-allylic position equivalent (BAPE) measures the number of single and double acting carbon sites in fatty acids respectively in fatty acids or esters having methylene interrupted polyunsaturation. The general trend in the increasing stability of different fatty acids is Linolenic< linoleic< oleic [9].

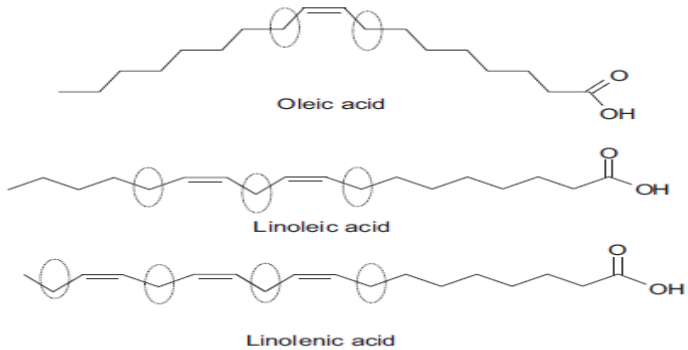


Fig. 1: Structure of Oleic acid, lenoleic acid and lenolenic acid [11].

IV. STABILITY OF BIODIESEL

Stability of biodiesel can be mainly classified into three types:

A. Oxidation stability
B. Thermal stability
C. Storage stability

Oxidation stability is related with the peroxides and hydroperoxides formation which further leads to formation of shorter compounds like acids (low molecular weight), alcohols, aldehyde and ketones [13]. Thermal stability is referred to the resistance offered by biodiesel to the oxidation process at the high temperature that leads to increase in weight of oil and biodiesel. At high temperature, natural antioxidants (very small quantity) present in biodiesel are degraded at much faster pace and leads to the prominent oxidation process. Storage stability deals with the degradation of biodiesel and its interaction with the surrounding conditions such as light, metal, moisture, impurities etc during the time of being storage. Storage stability is referred as the resistance shown by the biodiesel to the physical and chemical changes occurring in the composition of biodiesel during the storage due to its interaction with light, air, moisture and impurities [12].

V. OXIDATION DEGRADATION CHEMISTRY

Biodiesel is susceptible to oxidation when subjected to light, air, moisture etc., leading to formation of peroxides and hydroperoxides which further breaks into short chain compounds like acids, alcohols, aldehydes and esters. Biodiesel oxidation is of two types—auto-oxidation and photo-oxidation. Auto-oxidation occurs in presence of oxygen, while photo-oxidation occurs in presence of light and oxygen. The mechanism of degradation is as:

A. Autoxidation in presence of atmospheric oxygen
B. Thermal or thermal-oxidative degradation from excess heat
C. Hydrolysis in presence of moisture or water during storage and in fuel lines
D. Microbial contamination from impurities present in fuel [14].

The primary oxidative degradation of biodiesel is a radical chain reaction and involves initiation, propagation and termination step. During the initiation step, hydrogen gets removed from carbon radical and a carbon free radical is formed as shown in fig. These initiators may be formed either due to thermal decomposition of hydroperoxides or metal catalyzed decomposition of hydroperoxides [12]. During the propagation step, carbon free radical reacts with a bi-radical oxygen in air and forms peroxyl radical. This peroxyl radical further separates hydrogen atom from another biodiesel molecule and forms a hyroperoxide and another carbon free radical. Similarly the reaction propagates leading to formation of new radical at exponential rate. In the termination, the primary reaction ends by the combination of two or more radicals and forming a stable product [12].

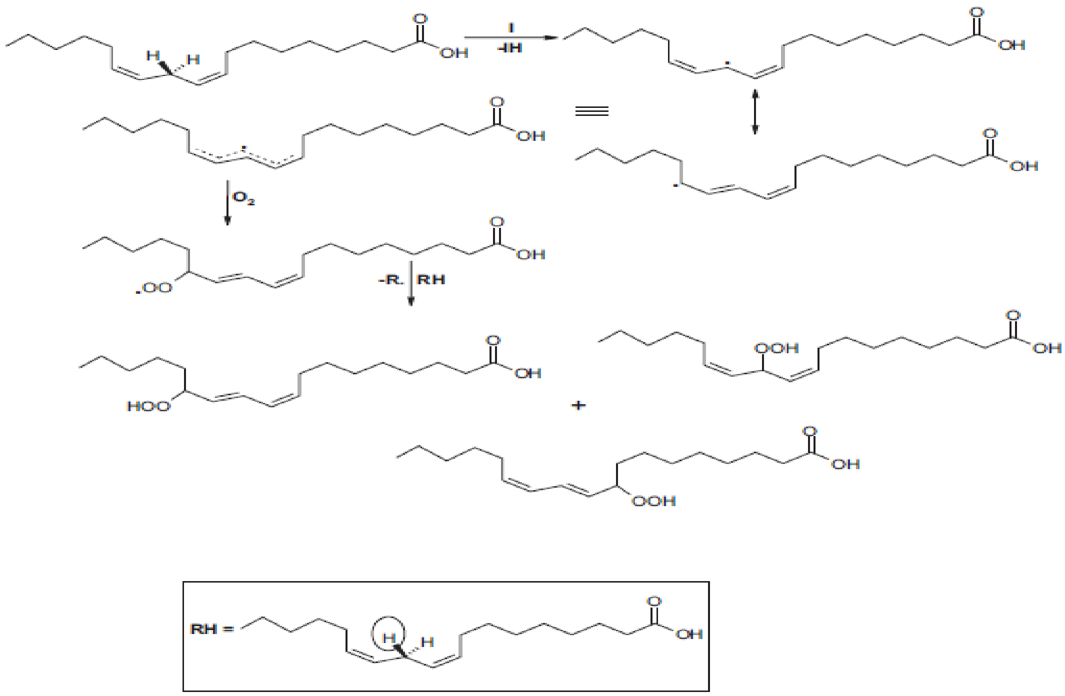


Fig. 2: Mechanism for auto-oxidation of lenolic acid methyl ester leading formation of its peroxides [11]

While in photo-oxidation the UV light decomposes the oxygen-containing compounds in biodiesel and generates radicals that initiate auto-oxidation. Photo-oxidation occurs via two routes. One similar to auto-oxidation, in second route molecular oxygen is excited to the state of electrophilic singlet and undergoes the ene reaction with olefins leading to the formation of hydroperoxides [11].

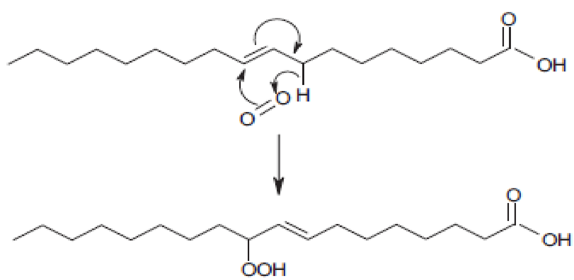


Fig. 3: ene reaction during photo-oxidation of linoleic acid [11]

For polyunsaturated fatty acids, the oxidative mechanism can be represented in three phases as shown in fig.4 [15]. Phase 1 is the phase of lag or phase of induction period, phase 2 is the phase of peroxidation. In phase 3 the rate of peroxide degradation exceeds the rate of peroxide formation.

In the induction period phase, the consumption of oxygen and formation of hydroperoxides occurs at a slow pace. Antioxidants are depleted at a very high pace in this phase yet the composition of biodiesel is significantly unaffected by oxidation process. Concentration of the antioxidants present in biodiesel mainly determines the length of this induction phase. In phase 2 i.e. phase of peroxide formation, oxygen consumption and hydroperoxides formation occur at faster pace than the induction phase. Antioxidants present in biodiesel are almost depleted in this phase. Finally in phase 3, rate of hydroperoxide degration is surpassed by the rate of peroxide formation leading to the exponential increase in the formation of small chain molecules such as acids, alcohols, aldehydes, ketones etc. Due to the formation of these small chain molecules the quality of fuel is highly degraded.

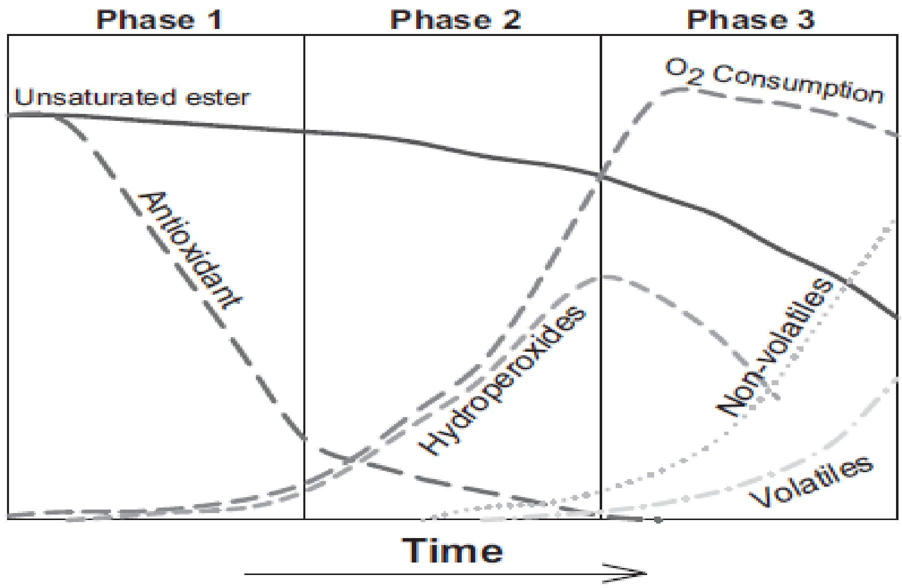


Fig. 4: Graphical representation of oxidation phases of polyunsaturated fatty acid esters [15]

VI. PARAMETERS INDICATING OXIDATION STABILITY OF BIODIESEL

As the biodiesel undergoes the oxidation process the quality of fuel is degraded. In order to measure the quality of fuel, various parameters are used which determines the extent of oxidation. The main parameters which are used to measure the stability of biodiesel are as follows:

A. IODINE VALUE (IV)

The acid value is the measure of the total degree of unsaturation present in the fatty acid compound irrespective of the nature and position of unsaturation. The Iodine value is expressed as the grams of iodine consumed per 100 grams of the sample [11]. The iodine value indicates the tendency of biodiesel to oxidize or polymerise depending on the reactivity of alkyl double bonds present. The iodine value does not differentiate in the nature, position and number of olefinic carbon present in fatty acid compound. Hence Iodine value does not determine the hydrogen as allylic or bis-alllylic. Since bis-allylic hydrogen are more prone to oxidation than the allylic ones [12]. So, it is not a very good parameter in determining the oxidation stability of biodiesel.

Table 1: Iodine value of various biodiesel samples [11]

Fat/Oil	Iodine Value (mg KOH/g oil)
Grape Seed Oil	124-143
Palm Oil	44-51
Olive Oil	80-88
Coconut Oil	7-10
Palm Kernel Oil	16-19
Cocoa Butter	35-40
Jojoba Oil	~80
Cottonseed Oil	100-117
Com Oil	109-133
Wheat Gram Oil	115-134
Sunflower Oil	125-144
Linseed Oil	136-178
Soybean Oil	120-136
Peanut Oil	84-105
Ricebran Oil	99
Margarine “light”	37.3
Margarine	86.1
Lard	57.6

Rape Oil	11.4
Crudefish Oil	108.5
Tung Oil	163.1
Beef Fat	46.9
Canola Oil	188-193

B. PEROXIDE VALUE (PV)

The peroxide value is measures as milli-equivalents of peroxide units per kg of the biodiesel sample [11]. The peroxide value is a measure of the peroxide units formed during the primary oxidation process. Since the peroxide value changes as the secondary oxidation process proceeds, so the peroxide value is not a suitable measure to determine the oxidation stability of biodiesel sample.

Table 2: variation of peroxide value (mg/kg) over a period of 6 months for different biodiesels [16-20]

Methyl Ester	0 month	3 months	6 months
JME	4.16	21.21	38.26
KOME	4.9	20.21	23.9
MOME	4.7	21.02	22
SME	3.8	36.5	-
SME$	3.8	55	-
RBOME	3.10	13.6	21.64

C. ACID VALUE

Acid value represents the quantity of free fatty acids present in the oil sample. Acid value is the amount of base (potassium hydroxide (KOH)) used to neutralize the 1 gram of the sample [12].

Table 3: Variation of acid value (mg KOH/g) over a period of 6 months for different biodiesels [16-20]

Methyl Ester	0 months	3 months	6 months
JME	0.15	0.3	0.45
JME*	0.44	0.78	1.1
RME	0.12	0.54	0.84
CME	0.19	0.20	0.32
SME	0.11	0.30	0.46
PME	2.54	2.87	-
COME	0.85	1.18	-
RSME	0.19	0.26	0.5
RBOME	0.09	0.14	0.17

D. OXIDISABILITY

Oxidisability is the another parameter used to determine the stability of biodiesel. Oxidisability is given by

$$OX=\frac{0.02(\%O)+(\%L)+2(\%Ln)}{100}$$

Where O refers to Oleic acid (18:1), L refers to linoleic acid (18:2) and Ln refers to linolenic acid (18:3).The Oxidisability is generally applied to those biodiesel which predominantly contains 18 carbon fatty acids [11].

E. VISCOSITY

Viscosity is the measure of the resistance offered to the flow of the fluid. Viscosity is also a measure to determine the oxidation stability of the biodiesel. The viscosity of biodiesel increases with the increase in the carbon chain length, degree of unsaturation in free fatty acids and its esters. The viscosity of *cis* double configuration is found to be less than the viscosity of *trans* configuration. The position of double has very minor effect on the viscosity of the sample [25].

Table 4: Variation of kinetic viscosity (cSt at 40°C) over a period of 6 months [16-18, 20-23]

Methyl Ester	0 month	3 months	6 months
JME	4.38	5.00	5.63
JME*	6.1	7.1	8.1
RME	4.6	5.95	6.5

CME	7.3	7.45	7.9
SME	4.3	5.6	6.05
PME	4.92	5.92	-
COME	3.67	4.72	-
KOME	5.01	5.19	5.41
MOME	4.85	5.05	5.15
RBOME	4.14	4.2	4.3

F. INDUCTION PERIOD

As the result of oxidation of biodiesel there is an increase in the concentration of the hydroperoxides and peroxides. The oxidation process continues to proceed until the oxygen is completely depleted or the conversion of fatty acids into small chain compounds is completely done. The length of time from the start of the oxidation and the time at which sudden increase in the oxidation rate is there is known as the Induction Period [11]. Induction period is one the main parameters which are used to determine the oxidation stability of the biodiesel. It is usually measured by Rancimat test or by Oxidation stability index (OSI).

Table 5: Specifications related to induction period in biodiesel standards of various countries [12]

S. No.	Biodiesel Standard	Country/Region	Test Methods	Limits (induction time, hrs(minimum))
1	ASTM D6751	USA	EN14112	3
2	EN14214	Europe	EN14112	6
3	Fuel Standard(Biodiesel), 2003	Australia	EN14112	6
4	ANP 255	Brazil	EN14112	6
5	SANS 1935	South Africa	EN14112	6
6	IS 15067	India	EN14112	6

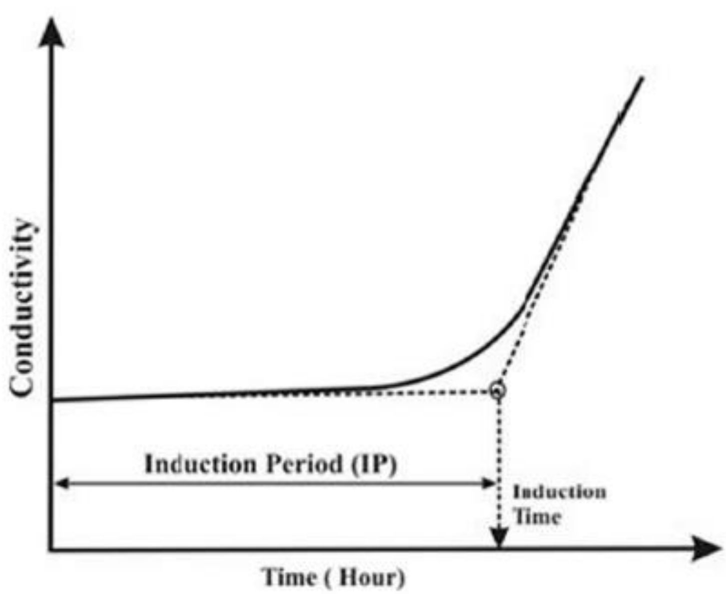


Fig. 5: Determination of Induction Period

G. STRUCTURE INDICES

The structure indices are one of the analytical indices which are used to measure the unsaturation of fats and fatty acid compounds. Allylic position equivalent(APE) and bis-allylic(BAPE) position equivalent are the structural indices which are used for the theoretical measurement of the oxidation stability of biodiesel or fatty acid compound.

Allylic position equivalent(APE) is the theoretical measurement of the number of allylic carbon present in the fatty acid compound assuming that all poly-olefinic unsaturation is methylene interrupted and bis-allyic position equivalent is the theoretical measurement of the number of bis-allylic carbon present in the fatty acid compound[25]. APE and BAPE are not so much useful measure in the estimation of the oxidation stability of the fatty acid compound because APE and BAPE do not consider the position of olenic carbon and bis-allylic olefinic carbon are more susceptible to oxidation than olefinic carbon.

The APE value of different fatty compounds can be calculated as[11]:
$APE=(ap_a \times A_{Ca})+(ap_b \times A_{Cb})+(ap_c \times A_{Cc})+\ldots$

Knothe suggested the following relation to determine the APE and BAPE values[25]:
$APE= 2\times(A_{C18:1}+A_{C18:2}+A_{C18:3})$
$BAPE=A_{C18:2}+2\times A_{C18:3}$
Where, A is the amount of each fatty acid compound and ap_x is the number of allylic position in fatty acid compound.

Knothe has also developed a correlation between Oxidation stability index (OSI) and BAPE as given below [25]:
$OSI=3.91 - 0.045\times BAPE \quad (R^2=0.983)$

H. DENSITY

Density is the measure of the mass per unit volume (Kg/m^3). It is also an important parameter which is used to measure the oxidation stability of the biodiesel. Due to the oxidation, a large number of small chain compounds are formed which leads to the increase in the density of the biodiesel. The fuels containing shorter chain hydrocarbon and more saturated fatty acid have more tendency to get crystallized. Due to crystallization, the volume decreases which leads to the increase in the density of the fuel [11].

Table 6: Variation of specific gravity over a period of 6 months for different biodiesels [17, 21-23]

Methyl Ester/Oil	0 month	3 months	6 months
JBD	0.85	-	1.9
JME	0.887	0.888	0.889
RME	0.888	0.890	0.891
CME	0.887	0.887	0.888
SME	0.887	0.888	0.889
PME	0.843	0.847	-
COME	0.844	0.856	-
RBOME	0.879	0.881	0.884

VII. FACTORS AFFECTING STABILITY

A. FATTY ACID STRUCTURE

Polyunsaturation present in the fatty acid compound make the compound susceptible to the oxidation. Along with the degree of unsaturation, the position of unsaturation in the compound also plays a very important factor in determining the oxidation stability of the fatty acid compound. Biodiesel with high degree of unsaturation and long carbon chains compounds degrade very quickly on storage.

B. RELATIVE ANTIOXIDANT CONTENT

Antioxidants are compounds whose presence in the oil and biodiesel make them less susceptible to oxidation. The antioxidants delays the oxidation process thus increases the stability of oil and biodiesel. Oils in their natural form contains the natural antioxidants which make the oil less susceptible to oxidation. On transesterification, during the distillation and purification process the natural antioxidants such as tocopherols, steols and tocotrienols get destroyed which make the biodiesel susceptible to oxidation [12].

On the basis of their existence antioxidants are generally classified as natural antioxidant and synthetic antioxidant. On the basis of the action antioxidants are classified as hydroperoxide decomposer and chain breaker. On the basis of the composition antioxidant are classified as phenolic and amine types. All the work related to stability is limited to the phenolic type of antioxidant and very scarce literature is available related to hydroperoxide decomposer type of antioxidant [9].

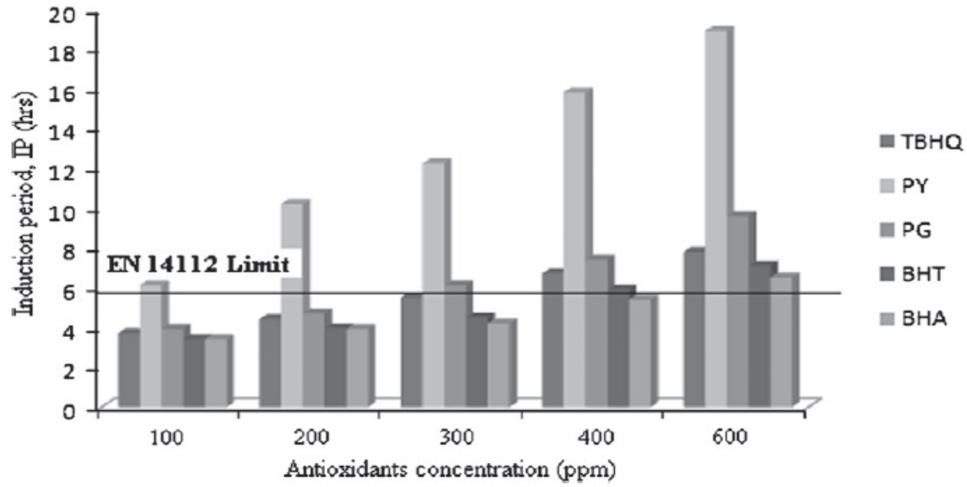


Fig. 6: Relative efficiency of the antioxidant on the jatropha biodiesel [9]

Oxidation stability of the biodiesel can be improved by adding the suitable antioxidant in the appropriate concentration. Antioxidant also helps in reduction of NO_x formation. The reason of increasing the stability with antioxidant addition is that highly reactive hydrogen present in the antioxidant gets readily separated by the peroxy radical than the fatty acid hydrogen, thus increasing the stability of the biodiesel.

C. METALS CONTAMINATION

It has been found that in various works that even the presence of small traces of the metals iron, copper, Nickel, cobalt, manganese etc. accelerates the process of oxidation[26,27]. The reason for the acceleration of oxidation process is that the metal enhances the catalytic reaction of the initiation of the oxidation [19, 26-29]. Non metals such as Viton, Teflon, Fluorinated plastics and Nylon are resistant to oxidation [30].

Table 7: Various antioxidants used for improving the stability [12]

S. No.	Type	Name	Abbrevation	Type	Nature
1	Natural	α-tocopherol	α-T	Phenolic	Chain inhibitor
2		β-carotene	β-C	Phenolic	Chain inhibitor
3	Synthetic	Butylated hydroxyanisole	BHA	Phenolic	Chain inhibitor
4		Butyl-4-hydorxytoluene	BHT	Phenolic	Chain inhibitor
5		2,5-di-tert-butyl-hydroquinone	DTHBQ	Phenolic	Chain inhibitor
6		tert-butyl-hydroquinone	TBHQ	Phenolic	Chain inhibitor
7		Propylgallate	PG	Phenolic	Chain inhibitor
8		Pyrogallol	PY	Phenolic	oxygen absorber
9		Hydrogenated cardanol	HC	Phenolic	oxygen absorber
10		Alkyl hydrogenated cardanol	AHC	Phenolic	oxygen absorber
11		Gallic acid	GA	Phenolic	Reducing agent
12		IONOX 220	-	Phenolic	Reducing agent
13		Vulkanox 2KF	-	Phenolic	Reducing agent
14		Baynox	-	Phenolic	Reducing agent

VIII. CONCLUSION

Biodiesel is the potential alternative to the conventional petroleum diesel but the property that is hindering in the commercial use of the biodiesel is the oxidation stability of the biodiesel. The stability is the ability to resist the physical and chemical changes due to interaction with air, light, moisture, impurities and on storage. The main factors affecting the oxidation stability are degree of unsaturation and the position of unsaturation. Acid value, peroxide value, induction period, viscosity, oxidizability, structural indices etc are the various parameters which are used to analyze the oxidation stability of the biodiesel. Fatty acid concentration, antioxidant concentration, metal contamination are major factors that influences the stability of the biodiesel. Synthetic antioxidants are used to increase the oxidation stability and majority of antioxidants are chain inhibitor. In this study article an attempt has been made to point out the factors affecting the stability of the biodiesel. There is the further scope in the field of the stability of the biodiesel which can ensure that the biodiesel can be made into the commercial use in daily life vehicles.

IX. ABBREVATIONS

CME	Castor Oil methyl ester
COME	Coconut Oil methyl ester
JBD	Jatropha Biodiesel
JME	Jatropha methyl ester
KOME	Karanja Oil methyl ester
PME	Palm Oil methyl ester
MOME	Mahua Oil methyl ester
RSME	Rapeseed Oil methyl ester
SME	Soybean Oil methyl ester in presence of carbon steel
SME$^{\$}$	Soybean Oil methyl ester in presence of galvanized steel

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X. REFERENCES

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