

Effect of synthetic cold flow additives on properties of biodiesel and biodiesel-diesel blends: A mini-review

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The demand for an alternative fuel to control fossil fuels is increasing, and biodiesel can play a crucial role in combatting the problems. Biodiesel that has been produced through various feedstocks employing different processes is a renewable and eco-friendly fuel. However, the generation of fewer emissions than fossil fuel makes it one favourable choice, but its marketability is still restricted due to poor cold flow behaviour and oxidation stability. In this review, we have focused on the cold flow problem of biodiesel which hampers functioning of diesel engines. This review particularly explores the types of synthetic additives, their mechanism of action, and their application in biodiesel-diesel blends in the last seven years.

Keywords: Biodiesel, Cold flow additives, Biodiesel-diesel blend, Polymer, Performance

Introduction

The need for biodiesel for diesel demand replacement is increasing because of lesser energy options in the transport, domestic and industrial sectors. Biodiesel has been produced from various methods like homogeneous, and heterogeneous catalysis, in-situ methods and other biotechnological routes which will mitigate GHG emission, providing a boost to energy sectors following sustainability goals.

Although biodiesel is a potential alternative to fossil fuel in terms of energy security, economics and environmental effect, it has drawbacks like poor cold flow properties, poor oxidation stability causing maximum instabilities in engines in cold climatic conditions¹. Currently, the major focus is to improve biodiesel's physicochemical characteristics to maximize stabilization. The drawback pointed out for biodiesel is related to its chemical structure. Biodiesel has myristate, palmitate, stearate, oleate, linoleate and linolenate as main constituents in its compositions². The unsaturated chain makes it more susceptible to oxidative degradation, consequently reducing engine performance³⁻⁵. The other crucial properties are cold flow related, which are affected by saturated esters present in biodiesel. The saturated ester causes the formation of solids and agglomeration of crystals,

which chokes fuel pipes and filters, thus create problems in engine operation and filters^{5,6}. The higher saturates in the biodiesel composition crystallize at lower temperatures and make the biodiesel more viscous. The highly viscous biodiesel causes problems in the operation of the fuel injection system.

The cold flow properties decrease if saturated chains content is low in biodiesel, and the oxidative degradation increases if unsaturated fatty acid content is high. The cold flow stability issues can be circumvented using different blends. As these properties depend on the fatty acid composition, blends with desired chain compounds are used. The blending component includes kerosene, oil, petrodiesel and chemicals with biodiesel⁷. Oil with high unsaturated and saturated carbon chains can be used as blending components to improve cold flow properties. Diesel biodiesel blends have been reported to improve the lubricity properties⁸. Kerosene and diesel also have been used as properties improver⁹. Different commercially available chemicals have also been blended like ethanol, acetone, and ethers to improve biodiesel's overall stability.

The main disadvantage of biodiesel blends is the increased NO_x emissions and the need to replace motor parts like fuel filters, deposits and other

compatibility issues^{10,11}. To address these issues, additives are needed to improve fuel characteristics and performance. However, different additives are used in combination with biodiesel to improve the overall engine emissions and performance, but the primary factor is the cold flow stability of biodiesel itself. So, this mini-review only explores the effect of synthesized cold flow additives on thermal stability not their applications in engines. Therefore, keeping the scope limited, this mini-review comprehensively explores the research carried out on synthesized cold flow additives about biodiesel stability during the last seven years.

Cold flow additives

Cold flow properties are one of the biggest concerns while using marketing biodiesel and biodiesel-diesel blends. The cold flow characteristics are important for determining fuel stability. As the temperature goes down, the different carbons in carbon chain fuel make the crystalline lattice. This crystalline structure of particles causes the biodiesel gels that block the fuel filters and create many problems. Various Methods like blending with oil, petrodiesel, crystallization fractionation, and transesterification using branched-chain alcohol and chemical additives have been investigated to improve low-temperature fluidity.

Factors affecting cold flow properties in biodiesel

The cold flow behaviour largely depends upon the fatty acid compositions¹². Saturated acid shows high antioxidant activity, high lubricity, high cetane number and low NOx emissions. Moreover, they have a high crystallization temperature and high melting point, but biodiesel with high-saturated contents exhibits low cold flow behaviour. Biodiesel has both saturated and unsaturated esters. Unsaturated esters have a low melting point as Biodiesel has more unsaturated fatty acid content and reduces cold flow properties^{13, 14}. Cold flow properties are influenced by chain length, too, as the Long carbon chain, which includes palmitic, oleic and lignoceric acid, exhibits poor cold flow properties. Biodiesel with a more short carbon chain would possess better cold flow properties¹⁴. The branching also plays a vital role in determining the cold flow behaviour of biodiesel. It increases the thermodynamic force, which is required to lower the crystallization temperature¹⁵. The straight-chain packs easily result from which low cold flow properties¹⁶. Biodiesel generally has methyl or

ethyl as its alcohol partner in transesterification. As the branched alcohols like isopropyl and butyl are used, they show better cold flow properties in the same carbon atom chain¹⁷.

Biodiesel cold flow behaviour also depends upon the production methods and type of feedstocks used. Li *et al.* used the two methods, viz. transesterification and thermal cracking, for biodiesel production, and it was observed that the pour point of transesterified biodiesel increases¹⁸. In contrast, cold filter plugging point(CFPP) decreases as opposed to thermal cracking based biodiesel. Further, when residual parts like glycerine, diglycerides or alcohol are present in a small amount, they also influence the cold flow performance. Saturated monoglycerides and steryl glucosides are high melting constituents that seeds the triglycerides crystallizations¹⁹.

Mechanism of action

Paraffin wax or saturated esters present in diesel-biodiesel fuels, however desirable to improve the engine performance due to their high cetane number and lesser emissions but have low solubility, which causes poor cold flow properties. In the typical mechanism, the saturates or wax crystals in fuels begin to crystallize when cooled down. These crystals adhere to each other and make a giant crystal that clusters and cause the fuel to gel. When flow improvers are added, co-crystallize with the saturates, block crystal growth faces, and make many small nuclei rather than large crystals. Further, additives alter the crystal face, and overall they slow down the crystal's growth in fuels, which leads to good cold flow behaviour (Fig. 1)

Type of cold flow additives

As cold flow problems are sensitive to engine performance, distribution and operability, therefore different techniques viz. winterization technique, ozonation, blending are used in literature. There is

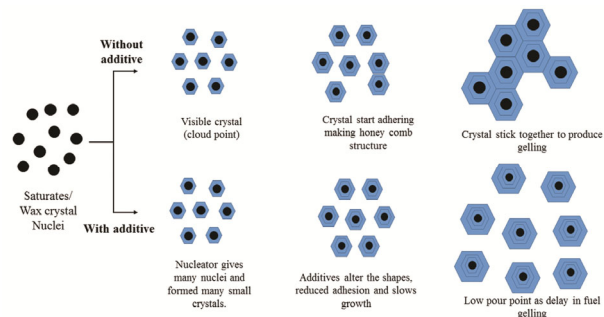


Fig.1 — Mechanism of Cold flow additive on biodiesel fuel

vast literature available in the aforementioned techniques. Herein, we focused on the synthesized chemical additives can improve cold flow behaviour. A different class of cold flow additives, viz. polymeric additive, alcohols, ethers, esters and metal-based additive, have been used in the literature, and some of them are shown in Table 1 and part of them are briefed in subsections.

Polymeric additives

Polymeric additives like poly-alpha-olefins, poly-(methyl acrylate) (PMA), EVAC have been used and have shown good improvement in the cold flow properties. In a study by Boshui *et al.*²⁰, three polymers viz. EVAC, PMA and OECP were taken

and mixed with soybean biodiesel. It was observed that OECP greatly enhance the cold flow properties even at small doses by providing a barrier to crystal agglomeration and inhibiting the wax crystal size.

Wang *et al.*²¹ used PMA, EVAC, PAO and poly-maleic anhydride on waste cooking oil and the addition of a small amount of PMA caused improvement in pour point (PP) and CFPP. In similar studies by Cao *et al.*²², the EVAC was used as a cold flow improver in UCO based biodiesel-diesel blends. As per their results, 0.04% concentration of EVAC in the B20 blend causes reduction of cloud point (CP), CFPP and PP by 8, 11, 10 °C, respectively. In a study by Verma *et al.*²³, EVAC and TGE was used as cold properties enhancers on palm biodiesel. As per their

Table 1 — Literature report on the different types of cold flow additives used in biodiesel

S. No	Type of additives	Additives used	Feedstock for Biodiesel	Inferences	Reference
1.	Polymeric additives	OECP, EACP, and PMA	Soybean	<ul style="list-style-type: none"> The addition of 0.03% of OECP reduces the PP and CFPP of biodiesel. 	[33]
		poly-alpha-olefin (PAO), EVAC, PMA, and poly maleic anhydride	Waste cooking oil	<ul style="list-style-type: none"> PMA found best, and the addition of 0.04% PMA decreased 8 °C and 6 °C PP and CFPP, respectively 	[34]
		Ethyl vinyl acetate copolymer (EVAC)	Waste cooking oil	<ul style="list-style-type: none"> The CP, CFPP and PP of the doped B100 biodiesel obtained 5, 2 and 1 °C, respectively. Reduction of 8, 10 and 11 °C was achieved for the CP, PP and CFPP of B20 	[35]
		Polyglycerol esters (PGE) and EVAC	Palm oil	<ul style="list-style-type: none"> The crystallization temperature of Biodiesel with EVAC, PGE and EVAC+PGE was reduced by 1.5, 3.5 and 3.1 °C, respectively. 	[36]
		poly(methyl acrylate)	B20 Coconut biodiesel	<ul style="list-style-type: none"> 0.03 wt % of PMA show the best cold flow properties. 	[37]
		Phenolic acid grafted with Ethylene-vinyl acetate (EVA)	B20 UCO biodiesel	<ul style="list-style-type: none"> At 0.1 wt% dosage of synthesized additive, the cloud point (CP), pour point (PP), and cold filter plugging point (CFPP) reduced by 5 °C, 10 °C, and 11 °C, respectively. 	[38]
		Fatty acid copolymer with glycidyl methacrylate	Esters from fatty acids	<ul style="list-style-type: none"> At 1000 ppm in all blends, the CP, CFPP and PP was reduced by 2, 8, 10 °C for B10 Blends. CP, CFPP and PP was reduced by 2, 13, 8 °C for B20 Blends. CP, CFPP and PP was reduced by 2, 11, 7 °C for B30 Blends. 	[39]
2.	Commercial chemicals	Ethanol	Pongamia biodiesel	<ul style="list-style-type: none"> The PP and CP of Biodiesel were reduced from 19 °C to 9 °C and 20 °C to 10 °C 	[24]
		ethanol, isopropanol and butanol	poultry fat methyl esters	<ul style="list-style-type: none"> Improved cold flow properties and the blending of butanol-PFME was better compared with ethanol and isopropanol. 	[42]
		DEP, PGE and PA,	Palm biodiesel	<ul style="list-style-type: none"> Different ratios of DEP: PGE: PA were used to provide the best effect. CFPP reduced by 7°C. 	[43]
		Butanol	Rapeseed oil	<ul style="list-style-type: none"> Improved properties of CP and CFPP of -16 and -31°C at 10% concentration in biodiesel. 	[44]

(Contd.)

Table 1 — Literature report on the different types of cold flow additives used in biodiesel

S. No	Type of additives	Additives used	Feedstock for Biodiesel	Inferences	Reference
3.	Esters	Ethyl levulinate	Cottonseed and poultry fat	<ul style="list-style-type: none"> The PP, CP, and CFPP of CSME were decreased to 3, 4, and 3 °C, respectively, The PP, CP, and CFPP were reduced to 4, 5, and 3 °C, for PFME, respectively. 	[45]
		Ethyl acetoacetate (EAA)	Waste cooking oil	<ul style="list-style-type: none"> Reductions of 4–5 °C in CP, 3–4 °C in PP and 3 °C in CFPP, respectively 	[46]
		Ethyl acetoacetate (EAA)/ Ethyl levulinate (EL)	Milk scum	<ul style="list-style-type: none"> CP, PP and CFPP of $8 \pm 0.33^\circ\text{C}$, 4 ± 0.57, and 7 ± 1.150 C were achieved with the EAA using 20% vol./vol. CP, PP and CFPP of 8 ± 0.33, 6 ± 0.66, and 7 ± 0.660 C were achieved with the EL using 20% vol./vol. 	[47]
		Methyl acetoacetate (MAA)	Waste cooking oil	<ul style="list-style-type: none"> 20 vol% MAA reduced CFPP, PP, and CP of WCO by 5 °C, 5 °C, and 7 °C. 	[48]
4.	Ethers	Branched ether	Soybean oil	<ul style="list-style-type: none"> 2-ethylhexyl ether was found suitable and decreased CP and PP by -23 °C and -25 °C, respectively 	[49]
5.	Alkoxy alcohol	Epoxidation and alkoxyated product	Canola oil	<ul style="list-style-type: none"> CP reduced by 6 °C. 	[50]
6.	Metal-based additive	Magnesium-based additive	Chicken fat methyl ester	<ul style="list-style-type: none"> The increase in additive concentrations to 16 l/mol/l into the chicken fat methyl ester caused a 7 °C decrease in Pour point. 	[51]
		Mn Additives	Pomace methyl ester	<ul style="list-style-type: none"> They found that doping the additive at a ratio of 12 l/mol/l with the methyl ester reduced the Pour point from 0 °C to -15 °C. 	[52]

results, the synergism of TGE and EVAC has shown significant improvement in crystallization temperature. Monirul *et al.*²⁴ used PMA in coconut biodiesel, and its B20 blends and addition of 0.03 % reduced the B20 blends' PP, CP and CFPP by 9, 3, 8 °C, respectively. Further, the PMA blended biodiesel was investigated for engine's performance and emission studies. As per the results, the 0.39% higher BSFC and 1.02% lower BTE were observed than baseline diesel. Furthermore, HC, CO and smoke opacity were lowered by 44.26%, 24.88% and 10.27%, respectively.

Han *et al.* synthesized the phenolic acids grafted ethylene-vinylacetate copolymers to improve the cold flow and oxidative stability of biodiesel-diesel blends²⁵ as reported in a recent study. As per the study 3,5-di-tert-butyl-4-hydroxybenzoic acid (DTBHA) and gallic acid (GA) were grafted on the ethylene-vinyl acetate (EVA) copolymers with different vinyl acetate amounts, and the grafted EVAs with gallic acid (EVA-GA-2) showed best result in the cold flow properties of B20 blends. Han *et al.* synthesized a novel comb-like copolymers were synthesized from higher fatty acids, para (p)-anisic acid, and glycidyl

methacrylate and checked the effect of biodiesel blends (B10, B20 and B30)²⁶.

Lie *et al.* conducted a study on the grafting of syringic acid (SA), 3,5-di-tert-butyl-4-hydroxybenzoic acid (DTBHA), and gallic acid (GA) onto polymethacrylate (PMA) copolymers, resulting in the synthesis of effective copolymers, namely PTG-SA, PTG-DTBHA, and PTG-GA. The results showed that the addition of 1500 ppm of PTG-GA to B20 (a blend of 20 vol% soybean biodiesel and 80 vol% diesel) reduced the CFPP and PP by 10°C and 18°C, respectively. Furthermore, the induction period (IP) of B20 was extended from 1.34 to 8.69 hours with the addition of 2000 ppm of PTG-GA²⁷.

Pucko *et al.* synthesized the polymeric additives incorporating methacrylate functional comonomers, namely, 2-(diethylamino)ethyl, 2-(diisopropylamino)ethyl, and 2-(tert-butylamino)ethyl methacrylate. These additives significantly enhanced low-temperature filterability, with the most notable improvement observed in the additive containing 2-(diethylamino)ethyl methacrylate (DEAEMA). This particular additive reduced the CFPP by 10°C and PP by 27°C²⁸.

N-containing compounds such as N-vinyl-2-pyrrolidinone (NVP), N-vinylimidazole (NVIM), and N-vinylcaprolactam (NVCL) also been coupled with longer chain of polymeric compounds to see the effect on biodiesel properties. Yang *et al.* prepared a series of such compound through free radical polymerization with C14-methacrylate (C14-MC). C14MC-NVIM (9:1) prepared biodiesel showed the highest Δ SP of 26°C and Δ CFPP of 13°C at 2000 ppm²⁹. In similar studies, they prepared alkyl methacrylate-norbornene anhydride copolymers (C14-methacrylate coupled with norbornene anhydride) and checked the influence on B30 biodiesel blends' cold flow properties³⁰. As per the results, at a dosage of 0.15 wt% , C14MC-NA-C14 showed the reduction in cold flow properties viz. CP, CFPP and PP of B30 by 5, 12 and 15 °C, respectively.

Commercial solvent as cold flow improvers

Various commercial solvents have been used in the literature to improve the cold flow properties. Sharma *et al.*³¹ used ethanol as a cold flow improver in Pongamia biodiesel, and a reduction of about 10°C was observed. Similarly, Joshi *et al.*³² used different types of alcohol, namely Ethanol, butanol, and isopropanol, on poultry fat methyl ester with three different concentrations of 5, 10 and 20%. The study found that butanol blended biodiesel show significant reductions in PP, CP and CFPP up to 7, 5 and 4 °C, respectively. In a similar study by Lapuerta *et al.*³³, butanol and ethanol were combined. Physicochemical properties were investigated and increased hexadecane value, heat value and lubricity of biodiesel were observed. Therefore, adding n-butanol into ethanol can prevent ethanol content from being too high to form a gel and extends to even higher butanol concentrations.

In the study lv *et al.*, three chemicals DEP, PGE and PA, were used on the palm methyl ester to improve the cold flow properties. However, the reduction of CFPP was observed when the additives' concentration was more than 1%, but there were no direct relationships between CFPP and fatty acid ester content³⁴. The addition of n-butanol solved biodiesel's instability at low temperatures and improved the flowability of biodiesel, which is supported by Makareviciene *et al.*³⁵. As per the study, rapeseed methyl ester (RME) and rapeseed butyl ester (RBE) was prepared, and various concentrations of butanol (10-100%) were added. It was observed that the butanol-RBE mixture show CP and CFPP of -16 and -

31°C at 10% concentration. A significant reduction in CFPP was observed when the additives were used in the synergetic ratio of 3:1:1 or 2:2:1. The plausible reason for the better cold flow properties may be forming hydrogen bonds between the hydroxyl group of alcohol and the ester group of biodiesels.

Synthetic additive as flow improvers

Some synthetic molecules have also been used to improve the cold flow properties. Joshi *et al.* prepared the ethyl levulinate and used it as a cold flow improver in cottonseed and poultry fat biodiesel³⁶. As per the reports, different concentrations of ethyl levulinate (2.5 -20 %) were used, and the reduction of PP, CP, and CFPP by 4-5, 3-4 and 3°C were observed at 20% concentration. Other physicochemical properties were also found in accordance with ASTM methods for \leq 15 vol % concentrations. The plausible reason for low cold flow properties may be due to ethyl levulinate's potential diluent nature, which can break the crystal formation at a temperature lower than room temperature, thus improving the fluidity.

Cao *et al.* used ethyl acetoacetate in UCO biodiesel to improve the properties³⁷. In similar studies, Srikanth *et al.* used both ethyl levulinate and ethyl acetoacetate in milk scum biodiesel, and a significant reduction in CP, PP and CFPP was observed³⁸. Similarly, methylacetoactate (MAA) was used as a diluent to enhance biodiesel's cold flow properties effectively³⁹. MAA in biodiesel altered the crystallization process, inhibit the agglomeration of crystals and reduced the amount of the crystal at low temperature, thus causing low PP, CP and CFPP.

Various branched ethers have also shown improvement in the cold flow behaviour of biodiesel. Moser *et al.*⁴⁰ studied the α -hydroxy ethers prepared from 9,10-epoxystearates using different linear and branched alcohols. As per their reports, 2-ethylhexyl alcohol was most promising for α -hydroxyl ethers generation and reduced the CP up to -26°C and PP up to -29°C for branched stearate.

Different branched alkyl esters have been explored in literature as the crystallization temperature of branched alkyl ester is lower than that of straight-chain head groups. Wang. *et al.*⁴¹ took canola methyl ester and synthesized branched-chain canola ester using 1-methoxy-2-propanol and 3-methyl-1-butanol. As per the results, the PP was reduced from -12°C to -27°C. Altaie *et al.*⁴² used palm-oil methyl ester (PME) and added PME and methyl oleate (MO) to diesel to make biodiesel blends. As the MO concentration

increased, the saturated methyl ester content in the blend decreased, and the palm-oil methyl ester content decreased, consequently improving the cooling fluidity of biodiesel. The low cold flow properties may be due to the high melting point of saturated long-chain fatty acids.

Metal or metal oxide has been investigated to improve biodiesel's cold flow properties^{43, 44}. As for biodiesel's different studies, the metal content should not exceed the limits explained by ASTM/BIS/EN methods. The fuel without the addition of pour point additive causes various problems in engines. The addition of a pour point additive prevents crystal accumulation and helps to improve engine performance.

As pour point additives directly do not affect engine performance and emission characteristics, these studies are omitted.

Recent advances in chemically synthesized Cold flow additives

The surfactant or biosurfactant has been used as a cold flow improver in recent literature as they have good surface activity and low critical micelle concentration. Further, they can reduce the wax crystal size and help in wax dispersion, which improves diesel flowability^{45,46}.

Nanomaterials have been widely used in industry with the evolution of nanotechnology in recent years⁴⁷. Due to nanoparticles' sizeable specific surface area, the heterogeneous nucleation mechanism is proposed to explain the appropriate modification of the nanoparticles' surface to enhance the interaction with the polymer PPDs⁴⁸. Graphene oxide (GO) can be used as nanohybrid materials to find better nanomaterials to improve the cold flow properties as graphene oxide have exfoliated structure, and strong disperse ability and good oil solubility⁴⁹.

Oils Resin also have shown significant improvement in cold flow properties. Zhang *et al.* prepared terpene resin from turpentine oil and checked the flow properties on soybean diesel-biodiesel blends. Even at concentration of 2000ppm, the CFPP got reduced by 10 °C for B30 blends⁵⁰.

One of recent advance to improve the biodiesel-diesel cold flow properties is to do winterization and additive introduction into prepared biodiesel. As per recent study by Singh *et al.*, the Karanja biodiesel was winterized and then plant based phenol additive was added to improve the cold flow properties. The combined effect shown great reduction of 7 (CP) and 6 °C (PP) at 1000 ppm⁵¹.

Conclusion and perspective

In the conclusion, various types of CFA were discussed, and reaction mechanism was explored. Further, the latest research about the application of CFA was critically reviewed with developed additives. A recent advancement of CFA was explored for future applications. In perspective, still more CFA can be synthesized as potential additives, which can significantly improve the quality of biodiesel. Further studies are required to investigate the enhancement of biodiesel cold flow behaviour, using chemically synthesized and their synergism petroleum diesel fuel or biodiesel may enhance the engine performance.

From a future perspective, cold flow additives are expected to play an increasingly vital role in enhancing fuel performance in low-temperature conditions. These additives will likely advance in improving the cold filter plugging point and pour point of fuels, ensuring better fuel flow and engine reliability during cold weather. As fuel formulations evolve and new engine technologies emerge, cold flow additives will offer greater adaptability and customization, catering to a wider range of fuel types and operating environments. Additionally, in the transition towards alternative fuels, cold flow additives will be crucial in maintaining fuel performance and stability, supporting the broader adoption of cleaner and more sustainable energy sources in cold climates.

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