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Terpolymers of alkyl methacrylate-trans anethole-1,2,3,6-tetrahydrophthalic anhydride copolymers: A low dosage and high-efficiency cold flow improver for diesel fuel

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A B S T R A C T

The addition of cold flow improvers (CFIs) is considered as the optimum strategy to improve the cold flow properties (CFPs) of diesel fuels, but this strategy is always limited by the required large dosage. To obtain low-dosage and high-efficiency CFIs for diesel, 1,2,3,6-tetrahydrophthalic anhydride (THPA) was introduced as a third and polar monomer to enhance the depressive effects of alkyl methacrylate-trans anethole copolymers (C_{14}MC-TA). The terpolymers of alkyl methacrylate-trans anethole-1,2,3,6-tetrahydrophthalic anhydride (C_{14}MC-TA-THPA) were synthesized and compared with the binary copolymers of C_{14}MC-TA and alkyl methacrylate-1,2,3,6-tetrahydrophthalic anhydride (C_{14}MC-THPA). Results showed that C_{14}MC -THPA achieved the best depressive effects on the cold filter plugging point (CFPP) and solid point (SP) by 11 °C and 16 °C at a dosage of 1250 mg/L and monomer ratio of 6:1, while 1500 mg/L C_{14}MC-TA (1:1) reached the optimal depressive effects on the CFPP and SP by 12 °C and 18 °C. THPA introduction significantly improved the depressive effects of C_{14}MC-TA. Lower dosages of C_{14}MC-TA-THPA in diesel exerted better improvement effects on the CFPP and SP than that of C_{14}MC-TA and C_{14}MC-THPA. When the monomer ratio and dosage were 6:0.6:0.4 and 1000 mg/L, the improvement effect of C_{14}MC-TA-THPA on diesel reached the optimum level, and the CFPP and SP were reduced by 13 °C and 19 °C, respectively. A 3D nonlinear surface diagram fitted by a mathematical model was also used for the first time to better understand the relationships of monomer ratios, dosages, and depressive effects of CFIs in diesel. Surface analysis results showed that C_{14}MC-TA-THPA achieved the optimum depressive effects at a monomer ratio of 6:0.66:0.34 and dosage of 1000 mg/L, and the CFPP and SP decreased by 14 °C and 19 °C, respectively. The predicted results were consistent with the actual ones. Additionally, the improvement mechanism of these copolymers in diesel was also explored.

As an important fossil energy source [1], the long-chain *n*-alkanes with carbon numbers between 9 and 22 in diesel are responsible for the poor cold flow properties (CFPs) of diesel fuel [2]. These long-chain n-alkanes in diesel tend to crystallize and accumulate [3], eventually forming larger wax crystals with three-dimensional (3D) network structures in cold regions [4]. These 3D structures can lead to the clogging of fuel pipelines of diesel engines, thus hindering their normal operation [5].

To overcome this cold flow difficulty, methods such as antifreeze technology [6], blending with coal-to-liquids [7], crystallization modifiers [8], and additives can be considered [9]. Among them, additives are recognized as some of the most cost-effective methods. Cold flow improvers (CFIs) are additives that effectively improve the CFPs of diesel [10]. SP refers to the lowest temperature at which diesel loses its flow properties, and CFPP represents the lowest temperature at which diesel passes through the screen of a diesel engine filter within 60 s [11]. CFI addition can significantly improve the cold cartridge performance of diesel fuel, including lowering the cold filter plugging point (CFPP) and solid point (SP) [12]. Polymethyl acrylate (PMA) is receiving considerable research attention because of its excellent structural diversity and ability to significantly improve CFPs [13]. Many PMA-based CFIs have been successfully developed as commercial CFIs (CCFIs) and are widely used to enhance the CFPs of diesel, biodiesel,

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or crude oils [14]. However, they are always limited by the required large dosages [15]. To develop better CFIs with low dosages and high efficiency, polarity and carbon-matching principles are extensively used to design and prepare new high-efficiency CFIs by modifying various polar/nonpolar groups and their monomer ratios [16]. Several studies have been performed in this respect [17]. Xie *et al.* [18]. synthesized a series of benzyl methacrylate–methacrylate copolymers (MB-R_1MC, $R_1 = C_{12}$, C_{14}, C_{16}, C_{18}) and benzyl methacrylate–tetradecyl methacrylate (MB-C_{14}MC) in different molar ratios of the functional monomers. They explored the effects of chain length on the CFPs of diesel. Results show that the presence of tetradecyl groups in CFIs of PMA systems is the most effective in improving the CFPs of diesel. The reduction in 3000 ppm MB-C_{14}MC (1:10) on the SP and CFPP of diesel is 26 °C and 12 °C, respectively. Chen *et al.* [2]. also synthesized a series of maleic anhydride–methyl benzyl acrylate co-polymer (MA-MB) and modified it with long-chain fatty amine and long-chain fatty alcohol (RNH_2 and ROH, $R = C_{14}$, C_{16}, C_{18}). They found that CFPP decreases by 8 °C when the amount of C_{14}MA-MB copolymer is 500 ppm. Therefore, the appropriate side-chain lengths can obviously enhance the depressive performance of CFIs. Generally, single polar and nonpolar monomers do not always produce beneficial effects, so third polar monomers are often introduced into the PMA polymeric system to solve such issues. A series of PMA of tetradecyl methacrylate-*N*-benzylmaleimide (C_{14}MC-BMI), tetradecyl methacrylate-4-acryloylmorpholine copolymers (C_{14}MC-ACM), and C_{14}MC-BMI-ACM has been synthesized by Yang *et al.* [19]. They comparatively studied the depressive effects of these binary and ternary polymers. Discussions have also been made about the synergy between the different nitrogen-containing parts contained in the trimers to improve the CFPs of diesel by adding a third polar group to these binary polymers as CFIs. Results show that adding the third polar part to the binary polymer can further enhance the performance of CFIs by adjusting the ratio between the parts.

In our previous work, trans anethole (TA) has been successfully extracted from star anise, a naturally renewable substance [20]. A novel natural CFI of alkyl methacrylate-TA copolymers (C_{14}MC-TA) were prepared to improve the CFPs of diesel. C_{14}MC-TA reduced the CFPP of diesel by 14 °C at a higher dosage of 2000 ppm. Nevertheless, the performance of C_{14}MC-TA was limited by its higher addition amount due to the structural particularity of the extract. Therefore, many different monomers based on PMA systems were explored, and found that the depressive effect of PMA-type CFIs could be improved effectively by taking the acidic anhydride monomer as the polar part. Also, 1,2,3,6-tetrahydrophthalic anhydride (THPA) with large spatial position resistance was always used as a synthetic material for surfactants. This study attempts to introduce 1,2,3,6-tetrahydrophthalic anhydride (THPA) as the third monomer for preparing terpolymers of alkyl methacrylate-TA-1,2,3,6-tetrahydrophthalic anhydride copolymers (C_{14}MC-TA-THPA). Meanwhile, the depressive effects of C_{14}MC-TA-THPA were evaluated and compared with those of binary copolymers of C_{14}MC-TA and C_{14}MC-THPA. Finally, a ternary nonlinear surface diagram fitted by a mathematical model was used to better understand the contribution of monomer ratios and dosages of CFIs for their depressive effects.

The composition and physicochemical properties of diesel fuel used in this work are given in Fig. S1 and Table S1 (Supporting information). The synthesis steps of the CFIs of C_{14}MC-TA-THPA, C_{14}MC-TA, and C_{14}MC-THPA are shown in Figs. S2 and S3 (Supporting information), and their chemical structure and molecular-weight distribution are given in Figs. S4 and S5, and Table S2 (Supporting information). Diesel fuel treated with these CFIs were formulated at 250, 500, 1000, 1250, and 1500 mg/L by weight.

As shown in Fig. 1, Figs. S6 and S7 (Supporting information), the dosages and monomer ratios of CFIs greatly influenced the inhibition effect of diesel CFPP and SP. The depressive effect of C_{14}MC-TA and C_{14}MC-THPA were obviously lower than that of C_{14}MC-TA -THPA. For C_{14}MC-TA, the depressive effect generally increased with increased dosage. It obtained the optimum performance at a C_{14}MC:TA molar ratio of 1:1, and the CFPP and SP reduction (ΔCFPP and ΔSP) achieved the maximum of 12 °C and 18 °C at a dosage of 1500 mg/L. Conversely, the depressive effect of C_{14}MC-THPA leveled off after the initial increase with the dosage range of 250–750 mg/L and 750–1500 mg/L. The optimum depressive effect on ΔCFPP and ΔSP reached 11 and 15 °C at a molar ratio of 6:1 and a dosage of 750 mg/L. Significantly, although the depressive effect of C_{14}MC-THPA failed to achieve the optimum inhibition effect of C_{14}MC-TA, the optimum dosage of C_{14}MC-THPA was only half of C_{14}MC-TA, whereas the difference in ΔCFPP between them was just 1 °C. Therefore, the introduction of THPA in C_{14}MC-TA-THPA obviously decreased the dosage of CFIs and increased the depressive effects at lower dosages. When the molar ratio of C_{14}MC:TA:THPA was 6:0.6:0.4 and the dosage was 1000 mg/L, C_{14}MC-TA-THPA achieved the optimum depressive effect. CFPP and SP also decreased by 13 °C and 19 °C, respectively. In PMA-type CFIs, the presence of long-chain alkyl groups can effectively be eutectic with the wax crystals in diesel, thereby changing the growth of wax crystals [21]. Accordingly, the wax crystals in diesel fuel cannot form a 3D network structure at low temperatures. However, when the polymer contained an excess of long-chain alkyl groups, it led to an increase in cross-linking between the polymer and the paraffin wax after eutectic, resulting in a poorer inhibition of CFPP in diesel by CFIs [22]. Meanwhile, THPA and TA had larger spatial site resistance than C_{14}MC, and the synthesized polymers had certain spatial site resistance compared with long-chain alkyl groups. The synergistic effect of two different polar groups in the polymer at an appropriate molar ratio can effectively make the long-chain alkyl groups in the polymer eutectic with the wax crystals to produce a certain degree of repulsion. This phenomenon exerted a significant inhibitory effect on the CFPP of diesel.

In general, finding maxima by fitting multiple sample data to 3D surfaces is a highly efficient and scientific approach [23]. As shown in Fig. 2, the Levenberg–Marquardt (LM) optimization algo-

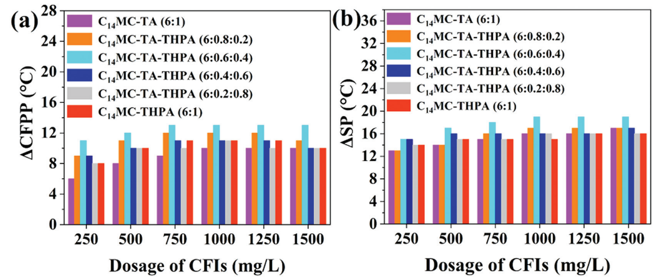

Fig. 1. Effects of various dosages and monomer ratios of C_{14}MC:TA:THPA on ΔCFPP (a) and ΔSP (b) of diesel.

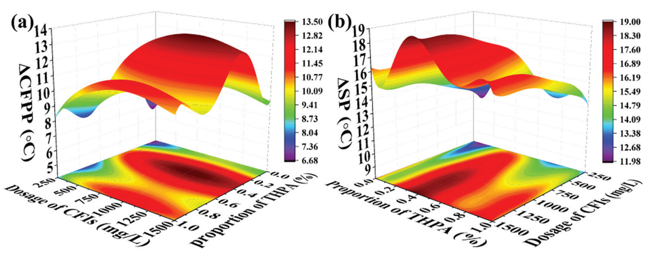

Fig. 2. Response surface analysis of the CFIs dosages and mononer rations effect of on the CFPP (a) and SP (b) of diesel fuel.

rithm was used, whereas the independent and dependent variables were fitted using the polynomial model. The fitting equation of 3D surface model is shown in Eq. 1, where Z is the height of a point from the surface, X and Y are coordinate points on the surface, Z_0, A_{1-5} and B_{1-5} are fitting parameters. By determining the fitting parameters, the optimal fitting surface is obtained, the residual difference between the fitting surface and the original data is minimized, finally the optimal solution is obtained. In this model, the molar ratio between the non-polar and polar part is 6:1, while the independent variable X denotes the molar ratio of THPA in the polar part (TA and THPA), the independent variable Y denotes the amount of CFIs added, and the dependent variable Z denotes the value of the change in diesel CFPP and SP. The CFPP and SP fitting parameters are shown in Tables S3 and S4 (Supporting information), respectively, where σ is the standard error and R^2 is the correlation. Notably, the fitted parameters R^2 were all close to 1, indicating that the effectiveness of the model for predicting the contribution of the addition and the monomer ratio to the effect of CFIs was extremely significant. Thus, it was ideal for predicting the inhibition of diesel fuel by CFIs with only a small sample. Response surface analysis results showed that C_{14}MC-TA-THPA reached the optimum depressive effect reduced at 6:0.66:0.34 molar ratio and 1000 mg/L dosage. The CFPP and SP of diesel fuel were reduced by 14 °C and 19 °C, respectively. Significantly, there are two peaks of ΔCFPP with the increase of proportion of THPA in Fig. 2, the main cause of this phenomenon is that the relative quantity of between TA and THPA changed the polarity and solubility of C_{14}MC-TA-THPA, therby changing the CFPP by modifying the crystallization behaviors of paraffins and isoparaffins in diesel. With the proportion of THPA increased, the ΔCFPP was first rising and reached the first peak at the monomer ratio of 6:0.66:0.34 by the synergies produced from TA and THPA. Giving it a larger steric hindrance, the polarity of C_{14}MC-TA-THPA became large and the solubility in diesel got worse after the first peak. More larger crystals were formed and irregular distribution in diesel, ultimately the ΔCFPP began to fall. According to our previous work [20], C_{14}MC-TA only works in enhancing the CFPs of diesel at high dosages. Small proportion of TA in C_{14}MC-TA-THPA is difficult to produce a positive synergistic effect with THPA in improving the CFPs of diesel. When the ratio of THPA:TA exceeds a certain value, the ΔCFPP was determined by the THPA in C_{14}MC-TA-THPA. Therefore, the ΔCFPP presented a slight increase, and formed the second peak in Fig. 2.

Therefore, the C_{14}MC-TA-THPA (6:0.66:0.34) was also prepared based on the best predicting date from the 3D surface and compared with other reported CFIs and CCFIs (Fig. S8 in Supporting information). Result show that the prepared C_{14}MC-TA-THPA (6:0.66:0.34) exerted better depressive effects on the CFPs of diesel at lower dosages, and the optimum improvement effect on SP and CFPP were consistent with the results of response surface analysis. Therefore, considering cost and performance, C_{14}MC-TA-THPA with a molar ratio of 6:0.66:0.34 was considered to be the optimum low-dosage, high-efficiency CFI for diesel fuels.

$$Z = Z_0 + A_1X + A_2X^2 + A_3X^3 + A_4X^4 + A_5X^5 + B_1Y + B_2Y^2 + B_3Y^3 + B_4Y^4 + B_5Y^5. \quad (1)$$

To deeply understand the performance mechanism and the crystallization behavior is an essential precondition for the low dosage and high-efficiency CFIs for diesel fuel [24]. Polarizing optical microscopy (POM) can present the changes in quantity, size, shape, and crystal morphology before and after diesel treatment with CFIs [25]. Microscopy images of untreated diesel and diesel treated with various CFIs (1000 mg/L) at low temperatures are given in Fig. 3.

As shown in Fig. 3 and Fig. S11 (Supporting information), in untreated diesel, the number and sizes of wax crystals increased significantly with decreased temperature from −15 °C to −35 °C, and

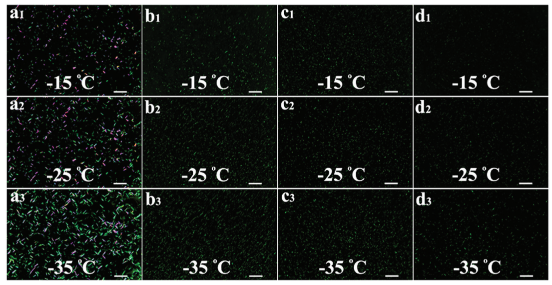

Fig. 3. Polarized optical micrographs at −15 °C, −25 °C, and −35 °C of untreated diesel (a_1, a_2, and a_3), diesel treated with 1000 mg/L C_{14}MC-TA (1:1) (b_1, b_2 and b_3), 1000 mg/L C_{14}MC-THPA (6:1) (c_1, c_2 and c_3) and 1000 mg/L C_{14}MC-TA-THPA (6:0.66:0.34) (d_1, d_2 and d_3). Scale bar: 100 μm.

3D network structures were formed by the cross-linking and stacking of these wax crystals at −35 °C. By comparison, the presence of various CFIs and CCFIs in diesel significantly modified the quantity, size, shape, and morphology of wax crystal. The prepared CFIs of C_{14}MC-TA, C_{14}MC-THPA, and C_{14}MC-TA-THPA in diesel exerted better improvement effect on reducing the quantity and sizes of crystals than the CCFIs of Hote and Nabren. Remarkable differences in quantity and sizes can be observed between C_{14}MC-TA, C_{14}MC-THPA, and the ternary polymer C_{14}MC-TA-THPA treated diesel. The wax crystals in C_{14}MC-TA-THPA treated diesel were considerably smaller and less than that of C_{14}MC-TA or C_{14}MC-THPA. The reason for this phenomenon was the presence of polar parts of THPA in C_{14}MC-TA-THPA generated a certain amount of spatial resistance, which destroyed the mutual cross-linking and aggregation of wax crystals and prevented the wax crystals forming into 3D network structures. Thus, the smaller number and sizes of wax crystals appeared at low temperature, but the liquid phase and crystals from passing through the filter and pipeline of engine cannot prevented. Consequently, the CFPP and SP of diesel significantly improved after diesel treatment with 1000 mg/L C_{14}MC-TA-THPA (6:0.66:0.34) (Fig. 3d). As shown in Fig. S12 (Supporting information), the wax crystal sizes of the microscopic images in the same region during the cooling process were calculated by ImageJ (image-processing software). With the decrease of temperature, the average size of diesel treated with C_{14}MC-TA-THPA was the smallest at each temperature. It was worth noting that the average size of diesel treated EVA12 and EVA18 was smaller than that of diesel treated Hote at −15 °C, and both gradually exceed Hote as the temperature decreases, which leads to the better depressive of CFPP and worse SP compared Hote as the temperature decreases.

For another, the macroscopic viscosity changes at different temperatures and the quantitative energy change and amount of precipitated wax crystals during the phase transition of diesel were explored by rheology and differential scanning calorimeter (DSC) in Figs. S9 and S10, and Table S5 (Supporting information). All these results were consistent with the conclusions for CFPP, SP, and POM.

The possible mechanism of the effect of C_{14}MC-TA-THPA on paraffin growth during diesel cooling is shown in Fig. 4. Without C_{14}MC-TA-THPA addition, diesel began to crystallize gradually at low temperatures. With further decreased temperature, paraffin waxes continued to deposit and grow on the initial nuclei, forming larger wax crystals, which cross-linked and adsorbed onto one another. Eventually, they formed a 3D network structure. The liquid phase of diesel was gradually wrapped by a large number of wax crystals with a 3D network structure, resulting in a gradual loss of diesel fuel fluidity at low temperatures. However, after adding C_{14}MC-TA-THPA to diesel, the paraffin in diesel preferred to cocrystallize with the long-chain alkyl groups in the polymer when the temperature started to decrease. Consequently, unstable small crystals were produced, which effectively retarded the growth of

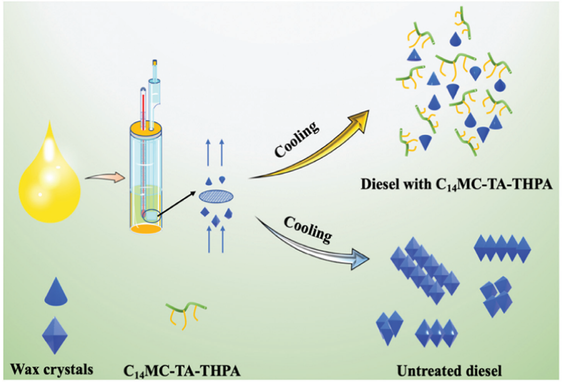

Fig. 4. Possible mechanism of C_{14}MC-TA-THPA for improving the flowability of diesel fuel.

wax crystals. The polar part of the polymer can effectively weaken the mutual attraction between the wax crystals and prevent them from cross-linking and adsorbing, which greatly reduced the possibility of paraffin waxes in diesel forming a 3D network structure at low temperatures. Ultimately, many small-sized wax crystals formed. These small and uniform wax crystals replaced the large-sized 3D network structure wax crystals, enabling diesel to show excellent CFPs and effectively reducing its CFPP and SP.

In summary, to obtain low dosage and high-efficiency CFIs for diesel, the terpolymers of alkyl methacrylate-TA-1,2,3,6-tetrahydrophthalic anhydride copolymers (C_{14}MC-TA-THPA) were synthesized and compared with the binary copolymers of C_{14}MC-TA and C_{14}MC-THPA. Results showed that THPA introduction significantly improved the depressive effects of C_{14}MC-TA. When the monomer ratio and dosage were 6:0.6:0.4 and 1000 mg/L, C_{14}MC-TA-THPA reduces the CFPP and SP of diesel by 13 °C and 19 °C, respectively. For the first time, the relationship among monomer ratio, dose, and CFI inhibition effect was better understood using 3D nonlinear surface diagrams fitted with mathematical models. Surface analysis results showed that C_{14}MC-TA-THPA achieved the optimum depressive effects at monomer ratios of 6:0.66:0.34 and dosage of 1000 mg/L, and CFPP and SP decreased by 14 °C and 19 °C, respectively. Therefore, the C_{14}MC-TA-THPA (6:0.66:0.34) was also prepared and the predicted results were consistent with the actual ones. Finally, through viscosity–temperature curve, DSC, and POM analysis, the action mechanism of C_{14}MC-TA-THPA in diesel was discussed in depth.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Bowen Xu: Data curation, Investigation, Methodology, Validation, Writing – original draft. **Jiahao Chen:** Investigation, Methodology. **Lulu Cui:** Investigation, Validation. **Xinyue Li:** Methodology, Validation, Visualization. **Yuan Xue:** Conceptualization, Data curation, Methodology, Resources, Supervision, Writing – review & editing. **Sheng Han:** Conceptualization, Funding acquisition, Project administration, Supervision.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2024.110196.

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