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REVIEWER'S REPORT

Manuscript No.: IJAR- 59136

Title: SYNTHESIS AND INVESTIGATION OF MODIFIED ACTIVE SILICA FILLERS,

Recommendation:
Accept after minor revision

Rating	Excel.	Good	Fair	Poor
Originality		✔		
Techn. Quality			✔	
Clarity			✔	
Significance	✔			

Reviewer's ID: Bilquees Hamza

Detailed Reviewer's Repor

The manuscript titled **"Synthesis and Investigation of Modified Active Silica Fillers"** presents a systematic approach to synthesizing two oleic acid–based ionic modifiers—**diethanolammonium oleate** and **tetraethylammonium oleate**—intended to reduce silicon(IV) oxide (SiO_2) nanoparticle agglomeration and improve compatibility with organic polymer matrices.

While the chemical optimization, yields, and basic physical characterizations are well-executed, the manuscript requires targeted revisions to strengthen its claims and clean up presentation errors.

Synthesis and Reaction Optimization

The authors investigate two synthetic routes to prepare ionic surfactants from oleic acid (OA):

- **Diethanolammonium Oleate (OA-DEA):** Synthesized via direct acid-base neutralization between oleic acid and diethanolamine at a 1:1 molar ratio. The optimal parameters were identified as $85–90^{\circ}\text{C}$ for 2.0–2.5 hours, yielding 98.55% product conversion with an acid value of $0.84 \pm 0.02\text{ mg KOH/g}$ ($0.423\text{ wt}\%$ unreacted oleic acid).
- **Tetraethylammonium Oleate:** Prepared via a two-step process: first, neutralizing oleic acid with NaOH at $70–80^{\circ}\text{C}$ (30–45 min), followed by an ion-exchange metathesis reaction with tetraethylammonium bromide (TEAB) at $50–55^{\circ}\text{C}$ (1.0–1.5 hours). Filtering out the precipitated NaBr in acetone yielded 97.90% chemical conversion and a 75.39% process mass yield.

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Critical Note: Acid-base neutralizations between carboxylic acids and secondary amines are usually rapid. Heating a secondary ammonium carboxylate salt like OA-DEA at $85–90^{\circ}\text{C}$ for over two hours carries a high risk of thermal dehydration into a covalent amide ($\text{N-alkyldiethanolamide}$). The authors should provide ^{1}H or ^{13}C NMR data to confirm that the product remains an ionic pair rather than an amide.

Spectroscopic and Thermal Characterization

- **FTIR Spectroscopy:** Spectral analysis confirms carboxylate ion (COO^-) formation. Diethanolammonium oleate shows asymmetric (ν_{as}) and symmetric (ν_s) stretching at 1594.63 cm^{-1} and 1404.18 cm^{-1} ($\Delta\nu = 190.45\text{ cm}^{-1}$). Tetraethylammonium oleate shows bands at 1556.55 cm^{-1} and 1423.47 cm^{-1} ($\Delta\nu = 133.08\text{ cm}^{-1}$), alongside strong aliphatic $-\text{CH}_2-$ stretches at 2920.23 cm^{-1} and 2850.79 cm^{-1}. The absence of a strong $\sim 1710\text{ cm}^{-1}$ free acid carbonyl band in the tetraethylammonium derivative confirms complete salt formation.
- **Thermal Behavior (TGA/DTA):** TGA shows that tetraethylammonium oleate loses 4.75% mass up to 160.66°C due to residual acetone and moisture. Main thermal decomposition occurs between 160.14°C and 316.05°C (69.32% mass loss), with a peak thermal breakdown effect at 254.55°C. A final 17.93% loss occurs up to 601.77°C, leaving an 8.00% residue. This indicates the modifier is thermally stable for processing below 160°C, but unsuitable for high-temperature melt compounding above 200°C.

Required Corrections and Improvements

- **Demonstrate Direct Impact on Silica:** The manuscript focuses almost entirely on synthesizing the organic modifiers, yet the title promises modified active silica fillers. The authors must include data on treated SiO_2 particles—such as contact angle measurements, dispersion stability tests in nonpolar solvents, or surface area (BET) analysis—to prove the modifiers actually improve silica filler behavior.
- **Fix Typographical and Figure Artifacts:** Line 238 contains a typo (Stage !: instead of Stage I:). Figures 5 and 6 captions contain distorted text artifacts from system file paths (e.g., D メネハー・ヤ Allanov AVOlein...). These need to be cleaned up.
- **Correct Reference Encoding:** Reference 7 (Line 451) suffers from severe formatting and character encoding issues where words are merged without spaces. This reference must be re-formatted into standard text.

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Recommendation: Accept with Minor Revisions. The synthetic work and characterization are solid, but addressing the side-reaction risk, cleaning up formatting errors, and providing basic data on treated silica will make the paper much stronger