SYNTHESIS OF NANOMATERIAL FROM HEAVY FRACTION OF CRUDE OIL

A
Thesis
Submitted to the
Atmiya University,
For the Degree
of

Doctor of Philosophy

in

Chemistry

by

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October-2025

Summary

Chapter-1: Crude Oil Stability and Compatibility Evaluation Using TLC-FID SARA: Correlating P-Value with Colloidal Insolubility Index

This chapter highlights the development and validation of a reliable TLC-FID method for accurate quantification of SARA fractions in crude oils. By introducing validated response coefficients, the study overcame key limitations of traditional approaches and demonstrated strong agreement with standard reference methods (ASTM D6560, IP 391, HTSD). Comparative analysis of synthetic and real crude samples established quantitative thresholds—CII < 1.0 and P-value > 1.45—as reliable indicators of crude oil compatibility. The findings also revealed that the asphaltene-to-resin ratio and API gravity are critical factors influencing crude stability. From an industrial perspective, the validated TLC-FID technique provides a rapid, cost-effective, and robust tool for predicting crude compatibility, optimizing blending operations, and reducing fouling risks. Overall, this work integrates methodological advancement with practical application, offering both analytical precision and significant operational benefits for refinery process optimization.

Chapter-2: Comprehensive Characterization of Aromatic, Resin, and Asphaltene Fractions Derived from Vacuum Residue

This chapter presents a comprehensive structural characterization of ARA fractions from diverse crude oil origins through the integrated application of FTIR, GPC, and NMR analyses. FTIR spectra confirmed the presence of multiple functional groups, including aliphatic and aromatic C–H, carbonyl, and heteroatom-linked functionalities, reflecting complex substitution patterns within aromatic domains. GPC data revealed that asphaltenes exhibit the highest molecular weights and broad polydispersity, particularly in HCO fractions, signifying pronounced structural complexity and aggregation tendencies. ^{1}H NMR analysis demonstrated a progressive decrease in aromatic and an increase in aliphatic protons from light to heavy fractions, while ^{13}C NMR confirmed enhanced aromatic carbon content and heteroatom incorporation in heavier fractions. Collectively, these results highlight the increasing chemical heterogeneity and structural intricacy of

heavier fractions, directly impacting asphaltene precipitation, fuel quality, and refining challenges. The complementary spectroscopic–chromatographic framework thus provides valuable insights into managing ARA fraction behaviour in refining processes.

Chapter-3: Preparation of Graphitic Carbon Like Material from ARA Fractions of Vacuum Residue

This chapter elucidates the synthesis and structural evolution of carbon materials derived from petroleum-based aromatic, resin, and asphaltene fractions via a controlled three-stage thermal treatment—green, calcined, and graphitized. Progressive pyrolysis facilitated near-complete carbonization, achieving yields exceeding 90% and ultimate carbon purities of 99.99%, as verified by elemental analysis. XRD patterns demonstrated a definitive transition from amorphous carbon to a crystalline graphite phase, marked by decreased interlayer spacing (from ~3.5 Å to ~3.37 Å) and increased crystallite dimensions (Lc and La). Raman spectroscopy corroborated this structural ordering through a pronounced reduction in the ID/IG ratio post-graphitization. Thermogravimetric analysis confirmed superior thermal stability of the graphitized products, exhibiting negligible mass loss. The convergence of compositional and structural properties across all fractions underscores the efficacy of the thermal conversion process in overcoming feedstock variability, thereby establishing a consistent and scalable pathway for producing high-purity, graphitic carbon from diverse petroleum precursors.

Chapter-4: Synthesis of Graphene Oxide and Reduce Graphene Oxide from Vacuum Gas Oil Derived Graphitic Carbon

This chapter presents a comprehensive comparative analysis of graphite, graphene oxide (GO), thermally reduced graphene oxide (rGO), and microwave-assisted reduced graphene oxide (rGO-M), establishing microwave-assisted reduction as a superior synthesis route for high-quality graphene-based materials. XRD and HRTEM analyses confirmed that rGO-M achieved near-complete structural restoration, with interlayer spacing (3.3960 Å) approaching that of pristine graphite (3.3858 Å). Elemental analysis and FTIR indicated

an exceptionally high C/O ratio (310.26) for rGO-M, evidencing extensive oxygen removal and sp^2 network recovery. TGA demonstrated its superior thermal stability, correlating with its high structural order. Morphological evaluation via FESEM and BET revealed rGO-M's exfoliated, mesoporous architecture, enhancing accessibility and mass transfer potential. Raman spectroscopy further validated the development of high-quality graphitic domains through a pronounced ID/IG ratio (1.52). Collectively, these results confirm that microwave-assisted reduction yields structurally superior, thermally stable, and functionally enhanced graphene materials suitable for advanced environmental and industrial applications.

Chapter-5: Reduce Graphene oxide applications in wastewater treatment and oil spillage

This chapter elucidates the mechanism and advantages of microwave-assisted reduction for the synthesis of reduced graphene oxide (rGO-M) from graphene oxide (GO). The process exploits the dielectric and polar nature of GO under 2.45 GHz microwave irradiation, enabling rapid, uniform, and energy-efficient deoxygenation. This selective excitation promotes the restoration of the conjugated sp^2 carbon network, thereby enhancing electrical conductivity, structural integrity, and material homogeneity. Compared to conventional thermal or chemical reduction methods, microwave-assisted reduction significantly minimizes defect formation and impurity incorporation while achieving superior control over surface morphology. The resulting rGO-M exhibits a high surface area, improved porosity, and excellent adsorption capability, making it particularly suitable for environmental remediation applications such as pollutant adsorption and oil spill recovery. Overall, the study demonstrates that microwave-assisted reduction provides a scalable and efficient route for producing high-quality graphene-based materials with enhanced physicochemical and functional properties.

Conclusion

Analytical Advancements

This study established a reliable analytical framework for crude oil characterization using the TLC-FID technique for precise SARA fraction quantification. The method, validated against standard procedures, effectively predicted crude oil stability and compatibility. Complementary FTIR, GPC, and NMR analyses provided molecular-level insights into the increasing aromaticity, heteroatom content, and structural complexity of heavier fractions, elucidating their influence on asphaltene behavior and refining performance.

Material Transformation

A three-stage thermal treatment—green, calcined, and graphitized—was successfully employed to convert petroleum-derived aromatic, resin, and asphaltene fractions into high-purity graphitic carbon. The process yielded over 90% carbon conversion with 99.99% carbon content. XRD and Raman spectroscopy confirmed the evolution from amorphous to ordered graphite structures, while TGA demonstrated exceptional thermal stability.

Advanced Graphene Development

Microwave-assisted reduction of graphene oxide (GO) produced reduced graphene oxide (rGO-M) with superior crystallinity, minimal defects, and enhanced porosity. The process achieved near-complete oxygen removal (C/O ratio ≈310) and interlayer spacing (3.396 Å) close to pristine graphite, outperforming conventional reduction methods.

Industrial Significance

Collectively, this research establishes a unified framework linking crude fraction characterization with advanced carbon material synthesis. The developed methodologies enable the efficient conversion of refinery-derived fractions into high-value, graphitic, and graphene-based materials. These outcomes advance sustainable refinery practices, offering scalable solutions for waste valorization and potential applications in environmental remediation, pollutant adsorption, and high-performance energy materials.