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Synthesis and Characterization of Oxide Catalysts Supported on Activated Carbon

B. A. Hadi[1], A. U. Muhammad[2], M.T. Umar[3]
Shehu Shagari College of Education, Nigeria[12]
Ibrahim Badamasi Babangida University, Nigeria[3]
Correspondent: alkanci123@gmail.com [1]

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ABSTRACT: The rising costs of conventional hydrotreating catalysts necessitate sustainable alternatives. Here, activated carbon derived from Empty Fruit Bunch (EFB) fibre, a byproduct of palm oil production, was developed as a renewable catalyst support. Using nickel nitrate, cobalt nitrate, and ammonium molybdate as both activating agents and precursors. NiO/C, CoO/C, and MoO_3/C catalysts were synthesized via in-situ activation. SEM/EDX analysis confirmed uniform metal oxide dispersion and revealed porous carbon structures. The results establish EFB-derived activated carbon as a low-cost catalysts support material with significant potential for catalytic upgrading of pyrolysis oil. Its high surface area and tunable properties further enhance its suitability for hydrotreating and other sustainable catalytic applications. This work introduces an in-situ route where metal precursors act as both activators and catalysts precursors, producing efficient EFB-derived catalyst supports for pyrolysis oil upgrading.

Keywords: Activated Carbon, Empty Fruit Bunch (EFB), Metal Oxide Catalysts, In-situ Activation, Hydrotreating, NiO/C, CoO/C, MoO_3/C, Porous Carbon, Sustainable Catalysis.

INTRODUCTION

Sub-Saharan Africa, with its extensive agricultural activities, generates vast quantities of underutilized biomass waste, representing a valuable resource for a bio-based economy (Rueda-Ordóñez et al., 2019). Oil palm (Elaeis guineensis), widely cultivated across the globe, traces its origins to West Africa and has a long history of domestication in Nigeria (Otti et al., 2014). Processing of fresh fruit bunches produces large amounts of biomass waste, including Empty Fruit Bunches (EFB), mesocarp fiber, palm kernel shell, palm kernel meal, and palm oil mill effluent, which could serve as feedstocks for sustainable applications (Elbersen et al., 2005). In Nigeria, despite the economic importance of the palm oil industry, the vast volumes of EFB generated pose significant environmental challenges, yet remain an abundant and inexpensive precursor for activated carbon production (Yoo et al., 2019). More broadly, agricultural residues lignocellulosic

materials generated from crop harvesting and processing are increasingly recognized as promising precursors for renewable energy production (Mortensen et al., 2011; Idris et al., 2016). These residues, categorized as field-based (primary) or process-based (secondary), hold considerable potential for thermochemical conversion into biofuels and bio-chemicals (Purnama, 2003). While direct combustion of biomass remains a simple energy option, particularly in rural Nigeria, its complex lignocellulosic composition offers far greater opportunities for advanced thermochemical applications.

The complex composition of lignocellulosic biomass presents opportunities for advanced thermochemical conversion processes, such as catalytic hydrodeoxygenation (HDO), which can yield higher-value sustainable hydrocarbons (Bai et al., 2019; Nachenius et al., 2013). Lignocellulosic biomass, the most abundant renewable source of organic carbon, is a central focus in the bio-based economy and is composed primarily of cellulose (40–50%), hemicelluloses (25–35%), and lignin (15–20%) (Centi et al., 2011; Anisuzzaman et al., 2015). This non-food resource can be deconstructed through thermochemical pathways into reactive intermediates for biofuel and biochemical production (Lindfors and Feiz, 2023; Triantafyllidis, 2013). The ongoing global energy crisis, driven by the increasing demand of modern society, underscores the urgent need to shift from finite fossil fuels major contributors to greenhouse gas emissions toward renewable alternatives such as agricultural residues (Hadi et al., 2017; Idris et al., 2021; Yaman, 2004).

Developing cost-effective and sustainable catalysts is critical for upgrading bio-oils. Activated carbon, with its high surface area and strong adsorption capacity, is widely recognized as an excellent catalyst support (Sun et al., 2012; Teo et al., 2016). However, commercial activated carbons are typically derived from non-renewable and costly raw materials. In contrast, producing activated carbon from waste agricultural products particularly oil palm biomass offers a more sustainable and economical alternative (Abdullah et al., 2011; Al-Harbi et al., 2018; Nb et al., 2016). High-surface-area activated carbons have been synthesized using chemical activating agents such as $ZnCl_2$, KOH, K_2CO_3, and H_3PO_4 (Muhammad et al., 2021; Triana et al., 2025). Among these, Empty Fruit Bunches (EFB) present distinct advantages as a precursor, simultaneously addressing environmental challenges linked to waste disposal and providing a low-cost feedstock for catalyst supports (Yang et al., 2014; Yusufu et al., 2012). Moreover, the unique porous structure and tunable surface chemistry of EFB-derived activated carbon enhance metal dispersion and catalytic activity, making it a promising material for bio-oil upgrading(Hadjar et al., 2016).

This research aims to valorize Empty Fruit Bunch (EFB) fibers, a major agricultural residue in Nigeria, by producing activated carbon as a support for novel NiO, CoO, and MoO_3 catalysts. These activated carbon–supported catalysts will be explored for their potential in upgrading bio-oil and vegetable oils through deoxygenation. The use of cost-effective, high-performance catalysts under lower operating pressures is critical for the economic viability of bio-oil upgrading (Amouzadeh et al., 2025). Therefore, this study focuses on the synthesis and characterization of NiO, CoO, and MoO_3 catalysts supported on EFB-derived activated carbon for catalytic deoxygenation of hydrocarbons in pyrolysis oil.

METHOD

Synthesis of Catalysts using Activated Carbon as Support

EFB fibers, collected from three palm oil processing plants in Benue State, Nigeria, were utilized as the precursor for activated carbon production (Abdullahi et al., 2024). The EFB fibers were initially washed extensively with deionized water to remove surface impurities, followed by drying in an oven at 105°C for 24 hours. A 20 wt% of NiO/C, MoO3/C and CoO/C catalysts were synthesized via the in-situ wet impregnation method. Stoichiometric amounts of Nickel Nitrate hexahydrate ($Ni(NO_3)_2 \cdot 6H_2O$) and Ammonium Molybdate hexahydrate ((NH4)6Mo7O24.4H2O) were dissolved for each in deionized water and heated at 90°C for 2hrs to facilitate the formation of the metal oxide precursors. The dried biomass was then subjected to in-situ activation and simultaneous doping with nickel nitrate, cobalt nitrate, and ammonium molybdate as activating agents as well as metal oxide precursors to produce NiO/C, CoO/C, and MoO_3/C catalysts respectively via an in-situ activation method solution. Following impregnation, the mixture was maintained in a water bath at 90°C for 2 hours under constant agitation (700rpm) to ensure thorough reagent absorption. The impregnated sample was subsequently dried in an oven at 110°C for 24 hours. Then, activation process was carried out in a muffle furnace at 550°C for 4 hours (Liu et al., 2016). To eliminate residual activating agent and ash, the resulting activated carbon was refluxed with deionized water for 3 hours, a process repeated until a neutral pH was achieved. Finally, the activated carbon was refluxed with a 0.1M nitric acid (HNO_3) solution for 1 hour to further remove any remaining heavy metals (Anisuzzaman et al., 2015).

SEM-EDX Analysis for Catalysts Characterization

Samples were pulverized to ~0.15 mm (100 mesh) using a jaw crusher, disc mill, and vibrating cup mill, then mounted on carbon tape-coated stubs. The SEM was calibrated and operated under optimized parameters (accelerating voltage, beam current, and resolution) after a stabilization period. Mounted samples were inserted into the chamber, focused, and imaged at magnifications ranging from ×500 to ×150,000. Acquired micrographs were transferred to the EDX system, where selected regions were analyzed for elemental composition (Shrestha, 2016).

RESULT AND DISCUSSION

EDX Analysis of CoO/C Catalyst

The SEM–EDX results (Table 1) identify cobalt (41.78 wt%) and carbon (12.75 wt%) as the dominant elements in the synthesized catalyst. The unusually high cobalt concentration confirms substantial incorporation of the active phase onto the carbon support. Reported Co/activated carbon (CoO/C) catalysts typically exhibit loadings in the range of 5–20 wt% (Suri, 2001; Tsoncheva et al., 2015). The significantly higher cobalt fraction in this study suggests enhanced surface enrichment or stronger metal support interactions. This improvement could translate into greater catalytic activity and stability under hydrotreating conditions (Bregante, 2020). The carbon

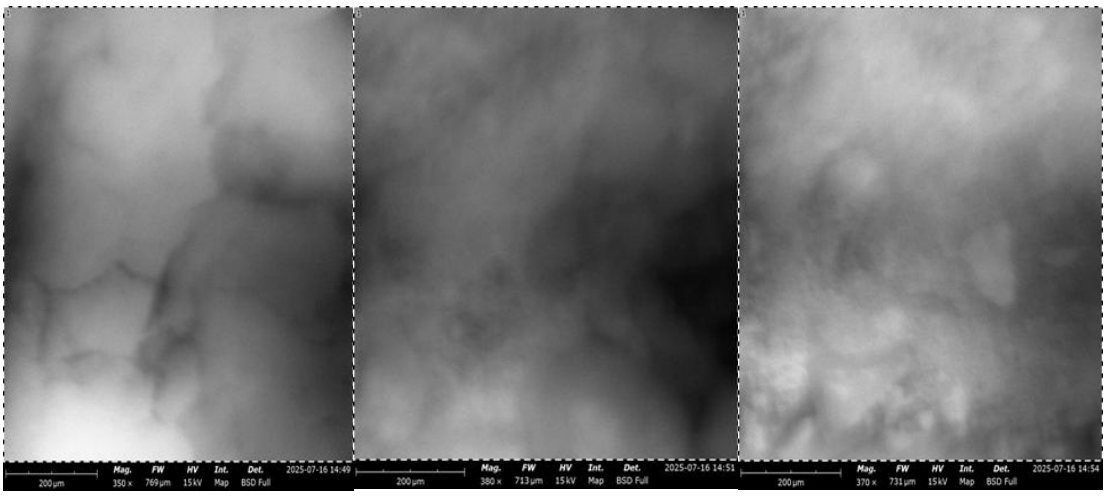
fraction (12.75 wt%) reflects the biomass-derived activated carbon matrix, which provides high surface area and anchoring sites for metal dispersion (B. Wang et al., 2023).

Table 1: EDX Analysis of CoO/C Catalyst

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
27	Co	Cobalt	22.50	41.78
6	C	Carbon	33.69	12.75
13	Al	Aluminium	13.94	11.85
14	Si	Silicon	8.36	7.40
20	Ca	Calcium	4.82	6.08
19	K	Potassium	4.86	5.99
12	Mg	Magnesium	4.27	3.27
26	Fe	Iron	1.83	3.22
16	S	Sulfur	1.92	1.94
15	P	Phosphorus	1.32	1.28
40	Zr	Zirconium	0.39	1.12
17	Cl	Chlorine	0.85	0.95
22	Ti	Titanium	0.44	0.66
41	Nb	Niobium	0.21	0.61
25	Mn	Manganese	0.27	0.48
29	Cu	Copper	0.22	0.44
23	V	Vanadium	0.12	0.19

SEM Morphological structural analysis in Plate1a revealed cobalt (41.78 wt%) and carbon (12.75 wt%) as the major constituents, confirming a high cobalt loading on a carbonaceous matrix. Reported CoO/C catalysts typically contain 5–20 wt% cobalt, such as ~7 wt% for ammonia decomposition and 2.5–15 wt% across CoO/C series (Wan et al., 2015). The significantly higher cobalt fraction observed in this study therefore places the material at the upper end of reported loadings suggesting Co-rich formulation. Such high loading is advantageous for catalytic activity, as it increases the density of active sites while the porous carbon support provides dispersion and stabilization.

Plate 1: SEM Image for the CoO/C (a), NiO/C (b) and MoO3 (c)



(a) (b) (c)

EDX Analysis of NiO/C Catalyst

SEM–EDX analyses presented in Table 2 of the cobalt and nickel-loaded activated carbon systems reveal strikingly different compositional features despite their shared biomass-derived carbon supports. By contrast, the NiO/C sample contained 37.04 wt% nickel and 19.41 wt% carbon, also surpassing the conventional NiO/C range (5–20 wt%) commonly applied in hydrogenation and reforming catalysis (Guo et al., 2019; Singh, 2017). Both systems therefore represent unusually metal-rich formulations, likely reflecting surface enrichment detectable by EDX, which could translate into high surface site density.

Table 2: EDX Analysis of NiO/C Catalyst

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
28	Ni	Nickel	17.70	37.04
14	Si	Silicon	19.64	19.67
6	C	Carbon	45.32	19.41
19	K	Potassium	3.09	4.31
20	Ca	Calcium	2.73	3.90
13	Al	Aluminium	3.83	3.69
26	Fe	Iron	1.43	2.86
12	Mg	Magnesium	2.14	1.86
16	S	Sulfur	1.37	1.57
40	Zr	Zirconium	0.46	1.49
50	Sn	Tin	0.34	1.43
56	Ba	Barium	0.17	0.84
15	P	Phosphorus	0.59	0.65
11	Na	Sodium	0.68	0.55
17	Cl	Chlorine	0.37	0.47
25	Mn	Manganese	0.14	0.27

SEM/EDX Characterization of MoO3/C Catalyst

Elemental analysis confirms molybdenum as the dominant element on the catalyst surface, at 42.48 wt%, which far exceeds the carbon fraction present at 20.29 wt%. This indicates a metal-rich surface composition that contrasts with typical MoO/C catalysts reported to have molybdenum loadings of 10–15 wt% (Ehweiner et al., 2021). Compared with CoO/C and NiO/C catalysts, which have lower metal fractions and higher carbon contents (41.78 wt% Co and 37.04 wt% Ni respectively), the MoO/C catalyst shows a notably higher enrichment of active metal species. The catalyst also contains significant amounts of aluminum (12.58 wt%) and silicon (9.64 wt%), elements attributed to its biomass precursor source, as well as alkali and alkaline-earth elements such as calcium (3.80 wt%) and potassium (3.62 wt%).

An important characteristic is the presence of sulfur at 3.22 wt%, unlike the negligible amounts found in the CoO/C and NiO/C catalysts. Sulfur presence is significant because, in Mo-based hydrotreating catalysts, sulfiding is intentionally introduced to form the catalytically active MoS_2 phase (Harsono et al., 2015; H. Wang et al., 2021). The naturally occurring sulfur in this catalyst may play a dual role where it could facilitate in-situ sulfiding and enhancing catalyst activation

(Zheng et al., 2023). Similarly, additional trace elements such as magnesium, copper, and zinc, all below 2 wt%, further emphasize the complex promoter profile of this biomass-derived activated carbon catalyst (Kun & Ksepko, 2025). Alternatively, it may pose risks of site blocking depending on operating conditions(Bai et al., 2019).

Taken together, SEM and EDX analyses portray the MoO_3/C system as a unique, metal-rich composite material with sulfur and promoter functionalities not as highly present in CoO/C or Ni/C catalysts (Sakidja, 2003). This composition suggests that while CoO/C and NiO/C catalysts may be more suitable for hydrodeoxygenation and hydrogenation, MoO_3/C catalysts could offer superior performance in hydrotreating or syngas upgrading applications where beneficial sulfur–molybdenum interactions are critical (Güvenatam et al., 2014; McMahon, 1992).

Table 3: EDX Analysis of MoO3/C Catalyst

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
42	Mo	Molybdenum	13.26	42.48
6	C	Carbon	50.59	20.29
13	Al	Aluminium	13.96	12.58
14	Si	Silicon	10.28	9.64
20	Ca	Calcium	2.84	3.80
19	K	Potassium	2.77	3.62
16	S	Sulfur	3.00	3.22
29	Cu	Copper	0.85	1.80
12	Mg	Magnesium	2.01	1.63
30	Zn	Zinc	0.43	0.94

Morphologically, the SEM micrograph of the Ni/AC sample (Plate 1b) illustrates a heterogeneous, porous surface typical of biomass-derived carbons, with micron-scale roughness that supports strong anchoring of Ni particles. Elemental analysis revealed secondary components including Al (11.85 wt%), Si (7.40 wt%), Ca (6.08 wt%), K (5.99 wt%), Mg (3.27 wt%), and Fe (3.22 wt%), consistent with the ash fingerprint of biomass-derived carbons. Rather than inert residues, these alkali and alkaline-earth species can act as in situ promoters, enhancing surface basicity, strengthening metal support interactions, and improving both activity and selectivity in hydrotreating and hydrodeoxygenation (HDO) pathways (Khandaker et al., 2025; Tsoncheva et al., 2015). In particular, K, Ca, and Mg are frequently reported to accelerate HDO rates and shift product distributions through base-catalyzed mechanisms (Adriano, 1986; Oudar, 1980). The unusually high silica fraction in the NiO/C sample suggests embedded SiO_2 domains that may impart acid–base bifunctionality and enhance thermal stability, as observed in Ni–SiO_2/C composites (Kwao et al., 2024). Trace elements such as S, P, Cl, Ti, Zr, and V were present below 1.5 wt% . While sulfur and chlorine are potential poisons, transition-metal oxides of Ti and Zr can contribute additional acid–base or redox functionality, potentially modifying catalyst performance (Bregante, 2020). Importantly, the much higher cobalt incorporation observed here compared to conventional CoO/C systems, when coupled with these intrinsic mineral promoters, suggests a synergistic effect (Gupta et al., 2021). This synergy is likely responsible for enhancing both catalytic stability and efficiency under hydrotreating conditions (Ay and Sen, 2021; Tsiotsias et al., 2020).

Table 4: Comparative physicochemical features of CoO/C, NiO/C, and MoO_3/C catalysts derived from activated carbon supports.

Catalyst	Major Metal (wt%)	Carbon (wt%)	Key Promoter Elements (wt%)	Notable Features and Implications
CoO/C	Co: 41.78	21.26	K (4.24), Ca (3.92), Si (8.27), Al (8.23), Fe (3.12), Mg (1.93), S (1.67)	Balanced metal/carbon ratio; high alkali and alkaline earth promoters enhance basicity; suitable for **hydrodeoxygenation (HDO)** and hydrogenation reactions.
NiO/C	Ni: 37.04	19.41	Si (19.67), Al (3.69), K (4.31), Ca (3.90), Fe (2.86), Mg (1.86), S (1.57), Zr/Sn/Ba traces	Significant Si/Al presence (from biomass ash) provides structural stability and acidity; Ni enhances **C–C hydrogenolysis**; good for hydrocracking and HDO.
MoO_3/C	Mo: 42.48	20.29	Al (12.58), Si (9.64), K (3.62), Ca (3.80), S (3.22), Mg (1.63), Cu/Zn traces	Strongly metal-rich surface; unique **sulfur co-presence** may facilitate in-situ MoS_2 formation, advantageous for hydrotreating and syngas upgrading.

CONCLUSION

This research has emphasized the promise of activated carbon-supported catalysts in hydrodeoxygenation and bio-oil upgrading, demonstrating how their high surface area, tunable porosity, and surface chemistry enhance catalytic activity. In particular, activated carbon derived from EFB not only offers advantages of abundance, low cost, and agricultural residue valorization but also provides a naturally promoted support matrix that enhances metal dispersion. The notably high metal content and the presence of intrinsic promoter elements in the EFB-derived activated carbon further distinguish this work, suggesting stronger metal support interactions and improved catalytic stability under hydrotreating conditions. While challenges remain including scale-up feasibility, reproducibility, and long-term stability future research should focus on in situ characterization of active sites, scalable synthesis strategies, and techno-economic as well as life-cycle assessments. Activated carbon-supported catalysts, especially those from EFB, represent a globally relevant and scalable pathway to green energy by aligning environmental sustainability with industrial innovation.

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