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Original Research Paper

Zinc nanocomposite from Cola acumulata husk extract: Synthesis, characterization and formulation into oilfield scale inhibitors

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Abstract

Scaling or solid deposits accumulation is a very serious challenge in oil and gas production systems as it blocks fluids flow, reduce production rate, damage equipment and increase maintenance cost. In order to mitigate scaling, a low cost, non-toxic, new material was synthesized by bio-reduction of zinc (II) ions using *Cola acumulata* husk extract (CAHE) at 30 – 50 °C. The new material was characterized by using some microscopic and spectroscopic techniques and evaluated as calcium scale inhibitor in oilfield environment. The new material was found to be zero-valent zinc nanocomposites with spherical shape and 76 - 83 nm average size. On evaluation using static scale jar method, the *Cola acumulata* husk extract-zinc nanoparticles (CAHE-ZnNPs) was 60.40 % efficient in inhibiting calcium carbonate scale at 30 °C but the efficiency decreased gradually to 49.8 % as temperature increased to 60 °C. On blending CAHE-ZnNPs with sodium pyrophosphate (Na-OPP) and ferric pyrophosphate (Fe-OPP), the scale inhibition efficiency at 30 °C increased to 74.1 % and 91.8 % but gradually decreased at 60 °C to 65.3 % and 82.8 % respectively. Another blend of CAHE-ZnNPs with both Na-OPP and Fe-OPP afforded efficiency of 97.2 % and 91.1% at 30 °C and 60 °C respectively. Inhibition efficiency increased with increase in dosage of CAHE-ZnNPs but slightly declined with increase in temperature. Optimal efficiency was achieved at 750 - 1000 ppm concentration of CAHE-ZnNPs. Thus, instead of discarding *Cola acumulata* husks as waste, it can be extracted, modified into this new material and utilized in mitigating oilfield scale deposition as reported here for the first time.

Keywords: Calcium carbonate scales, Oilfield chemicals, Scale inhibition, TEM, XRD, Zinc nanoparticles.

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1. Introduction

Scaling is the accumulation of solid deposits, precipitates or debris on the walls of metallic pipework during petroleum production and hydrocarbons recovery. Scales are usually formed in the wellbores, surface equipment and pipelines as crystals owing to fluctuations of thermodynamics, kinetics and changes in pressure and temperature values (Taha & Amani, 2019). Once deposited, their continuous growth results in reduction of the internal diameter of the production tubing or flow pipelines which may result in plug and reduction in production (Rostron, 2018 and Yousuf, et al., 2017). Therefore, scale formation can negatively impact the profitability of a production project if not managed properly. In oilfields, scales can be formed near wellbore, downhole completion/perforations or valves and separators mainly due to the presence of water (Taha & Amani,

2019). One of the key causes of scaling is the mixing of incompatible waters (Yang et al., 2020 and Ghalib et al., 2023). For instance, mixing of injection and formation waters may result in the precipitation, which is usually responsible for creating sulfate scales. If injection water that contains high amounts of sulfate is mixed with formation water that contains barium, strontium or calcium ions, different types of insoluble precipitates such as barium sulfate ($BaSO_4$), strontium sulfate ($SrSO_4$) and anhydrite ($CaSO_4$), will be deposited as scales. When incompatible water is in contact with formation rock, chemical reaction may occur and the insoluble species may be deposited as scales during its transportation due to temperature or pressure changes (Abbasi & Khamehchi, 2021).

Scales are mainly categorised into two types; sulfate scales or acid insoluble scales and carbonate scales or acid soluble scales (Kamal et al., 2018). Calcium carbonate and calcium sulphate are the main carbonate and sulfate scales often encountered in the field (Amiri & Moghadasi, 2012). Therefore, calcium scales are very crucial and worth attention. They have been managed by dissolution using acids, especially hydrochloric acid and sulphuric acid, and using chelants (Chauhan et al., 2015). Another way of managing scale has been to manipulate the fluid chemistry to make it non-scaling or manipulate thermodynamic conditions to prevent precipitation (Kumar et al., 2018). Alternatively, some chemicals called scale inhibitors can be injected to delay or slow down the rate of scale growth. The scale inhibitor would either adsorb onto the crystal surface to disrupt it further growth or adsorb on the steel surface to pervent crystals from adhering to the surface (Veloso et al., 2014 and Jafar Mazumder, 2020).

Several chemicals and formulations have been used as scale inhibitors, commonly phosphates esters, polyphosphates, organo-phosphorous compounds, poly-phosphono carboxylates and some chelating agents such as ethylene diamine tetraacetic acid (EDTA) (Ituen et al., 2021). Some of these compounds are expensive or cumbersome to synthesize in large quantity that is often deployed in the field. Ituen and coworkers (Ituen et al., 2017) had proposed that the use of plant materials could afford greener and cost-effective approach to designing scale inhibitors. Following this, some plant-based materials have been tested as alternative scale inhibitors (El Housse et al., 2024 and Cui et al., 2024). It has also been demonstrated that *Allium cepa* peels extract modified into silver nanoparticles can inhibit calcium scales better than crude plant extracts (Ituen et al., 2021). On this basis, the current research was designed. However, silver is known to exhibit some degree of toxicity, and the metal ion solution (silver nitrate) is very expensive and even banned in some countries. Therefore, we explored non-toxic metal ions sources (such as iron, zinc, copper, manganese, magnesium, etc) and heavy biomass for the synthesis, hence the choice of zinc and *Cola acumulata* husk, respectively. In this paper, preparation of zinc nanoparticles from CAHE, its nanostructure and surface characteristics as well as its performance as scale inhibitor without and with synergistic additives is reported.

2. Materials and Methods

2.1 Collection and preparation of Cola acuminata husk extract (CAHE)

Cola acuminata husks were obtained from Ette Market in Ikot Abasi Local Government Area of Akwa Ibom State and conveyed to our laboratory where they were cut into pieces, washed thrice in distilled water, dried at laboratory temperature and macerated (**Figure 1 a-c**). About 1000 g of the macerated samples were soaked in 10 L of distilled water and allowed for 24 h. Thereafter, it was filtered with muslin cloth, re-filtered using Whatman 1 mm filter paper and concentrated to dryness at 100 ^{o}C in water bath. The dry extract was mashed to powder using mortar and pestle, wrapped in aluminium foil, labelled as CAHE and stored in refrigerator prior to use.

2.2 Preparation of CAHE-ZnNPs

Analytical grade zinc chloride ($ZnCl_2$) supplied by Double Bond Chemicals, Uyo was prepared in de-ionized water to a concentration of 0.01 M and used for the synthesis. Freshly prepared metal salt solution was mixed with 1.0 g/L of CAHE at ratios (v/v) 1:1. The mixture was stirred periodically, maintained at 30 - 50 ^{o}C in water bath and observed for colour change (Al-darwesh et al., 2024). On colour change, the mixture was retrieved; one portion was dried in oven at 40 ^{o}C to powder, washed with de-ionized water, dried again at 40 ^{o}C and stored in

refrigerator; the other portion was stored in the liquid form, all for further analyses.

Figure 1. Cola acuminata husk after (a) collection (b) cutting into small sizes and (c) maceration.

2.3 Characterization of synthesized material

Aliquots of the liquid form of each of CAHE and CAHE-ZnNPs were diluted in de-ionized water and subjected to UV-Vis analyses at scan range between 200 - 800 nm Thermo Genesys 10 UV/Vis spectrophotometer. For the dried sample, a small portion was mixed with KBr and analysed by FTIR within the range of 600 - 4000 cm^{-1} using Agilent cary630 FTIR/ATR spectrophotometer. The crystallographic properties of the dried samples of both CAHE and CAHE-ZnNPs were also analyzed by XRD at $2\theta = 10^{o} – 80^{o}$ using PX1800 X-ray diffractometer with automatic divergence slit, a Cu anode producing X-rays of wavelengths $\lambda_1 = 1.54056 \times 10^{-10}$ m and $\lambda_2 = 1.54439 \times 10^{-10}$ m, operated at 40 kV and 55 mA. The elemental compositions of each of CAHE and CAHE-ZnNPs were also determined by EDS using Phenom ProX SEM/EDS spectrophotometer. The morphologies and sizes of each of CAHE-ZnNPs was probed by TEM using Fei Technai G2F20 microscope.

2.4 Scale inhibition experiment

Mild steel coupons obtained from Fortunate Steel & Ind. Mat. Co. Ltd, Port Harcourt, Nigeria was used for scale inhibition experiment. The coupons of sizes 4 cm x 4 cm, were degreased in absolute ethanol, abraded with silicon carbide paper abrasives of various grits, and finished to mirror surface. Thereafter, they were rinsed in acetone, wrapped in sealed waterproof material and stored in desiccator prior to use. The scaling medium was formulated and pre-treated using standards previously reported by Ituen and coworkers (Ituen et al., 2021). The initial concentration of Ca^{2+} ions in the scaling solution (SS) was determined by Atomic Absorption Spectroscopy (AAS) using 235ATS Buck Scientific.

The static jar test method was used to determine the inhibition efficiency following standard procedures provided by NACE TM0374-2007 with slight modification as previously described by Ituen and co-workers (Ituen et al., 2021). The concentration of Ca^{2+} ions (mg/L) in the inhibited solution was also determined by Atomic Absorption Spectrometry and used to calculate the scale inhibition efficiency (η) according to Eq. 1.

$$\eta = 100 \left(\frac{C_1 - C_2}{C_0 - C_1}\right) \quad (1)$$

where C_0 represents the initial mass concentration of Ca^{2+} ions (mg/L) in blank SS before immersion, C_1 and C_2 represent the mass concentrations of Ca^{2+} ions in SS with and without addition of CAHE and CAHE-ZnNPs respectively. The solution of Na-OPP and Fe-OPP were prepared to concentration of 1000 ppm and added to the CAHE-ZnNPs at the ratio 1:1 (v/v). The experiments were also repeated at 40 ^{o}C, 50 ^{o}C and 60 ^{o}C.

3. Results and Discussion

3.1 Formation and characteristics of CAHE-ZnNPs

The CAHE-ZnNPs developed and formed within 60 minutes, and this was signalled by change in colour of the mixture from yellowish-brown to dark brown as shown in **Figure 2**. The short formation time indicates that the

synthetic process is fast and feasible. The obtained colours are consistent with previously reported plant extract-based nanoparticles (Arif et al., 2023 and Iqbal et al., 2021). The change in colour resulted from bio-reduction of zinc ions to lower valency (Ituen et al., 2021).

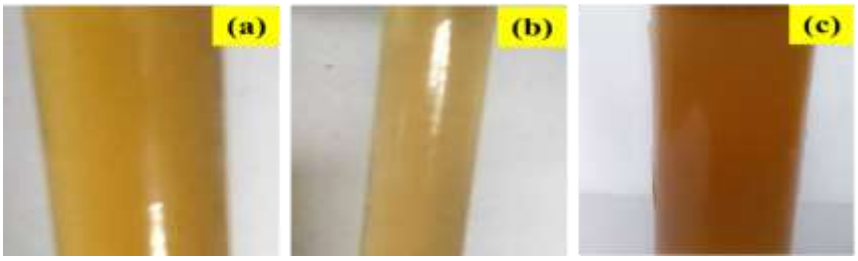

Figure 2. Colour of (a) CAHE (b) CAHE+ Zn salt at t = 0 and (c) CAHE + Zn salt at t = 60 min.

3.2 Microscopic analyses

TEM micrograph **(Figure 3a**) also revealed that CAHE-ZnNPs were near spherical in shape and of sizes between 76 - 83 nm, similar to literature report (Al-darwesh et al., 2024). From the figure, the particles were well dispersed, an indication of good stability against aggregation. Being within the nano range, the new material was therefore confirmed as nanoparticles.

3.3 Spectroscopic analyses

The UV-Vis absorption spectrum of CAHE and CAHE-ZnNPs obtained is shown in **Figure 3b**. The figure reveals that CAHE and CAHE-ZnNPs absorb at different wavelengths. Maximum absorption for CAHE was obtained at 366 nm whereas that of CAHE-ZnNPs was obtained at 303 nm. The value of absorption maxima for CAHE-ZnNPs obtained is similar to that reported in related literature (Al-darwesh et al., 2024). Plant extract mediated nanoparticles usually demonstrate surface plasmon resonance, and consequently UV–Vis absorption in region characteristic of their optoelectronic properties (Adrianto et al., 2022). The difference in absorption characteristics between CAHE and CAHE-ZnNPs demonstrates that a new material was actually formed, and that the new material is different from the starting material.

The crystal structure of CAHE-ZnNPs was confirmed by XRD. Results, as analysed using JCPDS card, showed major diffraction peaks (**Figure 3c**) at 2θ values mainly at 32° (100), 35° (101, and 50° (102) assigned to (100), (101) and (102) planes. The peak at 35^{o} exhibited the highest intensity indicating that the Zn atoms are oriented maximally within the (101) plane (Ituen et al., 2021) with cubic Zn^{0} phase.

The FTIR analysis was carried out to determine and compare the functional groups associated with the surface of CAHE and CAHE-ZnNPs and the obtained spectra is shown in **Figure 3d**. The spectra showed the presence of various prominent peaks in both materials and these peaks were matched against Sigma-Aldrich IR Table and Chart. Firstly, both CAHE and CAHE-ZnNPs showed peaks at similar wave numbers but with slight displacement in vibrational frequency. This indicates that both CAHE and CAHE-ZnNPs are materials from similar origin with slight modifications. The main peaks were obtained at 1148, 1670, 1995 and 3670 cm^{-1} and assigned to C-O (aliphatic ester), C=O (carbonyl), C-H (aromatic), and O-H (alcohol) functional groups, respectively. These functional groups are likely from phytocompounds such as kolatine, alkaloids, phenolics, essential oils, caffeine and nicotine reported to be abundant in CAHE (Jacob et al., 2024). The slight displacement in wave number of peaks of CAHE and CAHE-ZnNPs could signify that the associated functional group may have been involved in the bio-reduction reaction.

The EDS analysis was also conducted to check the elements on the surface of the materials and the various elements found on the surface as well as their compositions are displayed in **Table 1**. Results confirm that about 8.08 % of Zn was incorporated into the CAHE-ZnNPs matrix in addition to C, N and O that were capped from the organic compounds in CAHE.

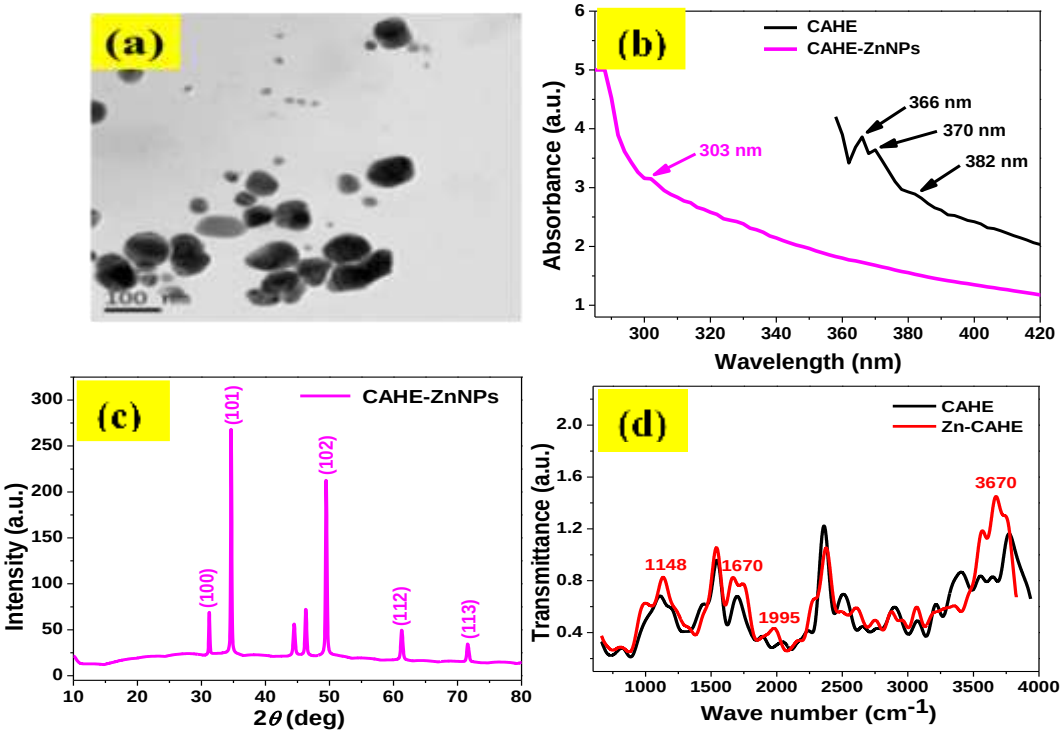

Figure 3. (a) TEM image of CAHE; (b)UV-Vis spectra of CAHE (black) and CAHE-ZnNPs (purple); XRD diffraction pattern of CAHE-ZnNPs; and (d) FTIR spectrum of CAHE (black) and CAHE-ZnNPs (red).

Table 1. Elemental composition of CAHE and CAHE-ZnNPs obtained from EDS study.

Element	Composition (wt. %) in CAHE	Composition (wt. %) in CAHE-ZnNPs
C	79.78	73.56
N	3.08	2.87
O	17.14	15.49
Zn	-	8.08

3.4 Scale inhibition

The inhibition efficiency of 250 ppm of both CAHE and CAHE-ZnNPs were determined at various temperatures (30 – 40 °C) and compared. It can be observed from the results obtained (**Table 2**) that at 30 °C, the inhibition efficiencies (%) of 250 ppm of both CAHE and CAHE-ZnNPs were 48.3 % and 60.4 %, respectively. While CAHE was not very efficient, its conversion to nanoparticles resulted in improved efficiency. However, as temperature was increased, the inhibition efficiency declined for both CAHE and CAHE-ZnNPs, which is characteristic of behaviours of many plant-based nanoparticles. The obtained efficiencies were not high enough and typical of plant materials. In attempt to improve the efficiency, the dose of CAHE-ZnNPs was increased and the obtained efficiency increased with increase in dosage (**Table 2**).

Table 2. Scale inhibition efficiency (%) of 250 ppm CAHE and different concentrations of CAHE-ZnNPs.

T (°C)	250 ppm CAHE	CAHE-ZnNPs			
		250 ppm	500 ppm	750 ppm	1000 ppm
30	48.3	60.4	63.4	64.5	68.8
40	45.9	58.2	60.9	62.2	65.0
50	37.6	55.1	54.3	56.7	59.4
60	28.4	49.8	51.6	52.3	54.1

On considering the desirable performance range for deployment in field (Liu et al, 2016), the observed value for the scale inhibition efficiencies of CAHE-ZnNPs may be considered low. It has been reported that a single compound or material may not be sufficiently efficient to be deployed in the field as scale inhibitor (Ituen et al., 2017). Thus, many commercially available scale inhibitors are composed of a mixture of many materials comprising of the major active ingredient, the synergistic intensifier, the solvent, and sometimes a surfactant. Therefore, to improve the efficiency obtained, 100 ppm of both CAHE and CAHE-ZnNPs were blended with sodium pyrophosphate (Na-OPP) and ferric pyrophosphate (Fe-OPP). The structure of Na-OPP and Fe-OPP is shown in **Figure 4**. Many commercial scale inhibitors contain long chain phosphates which may be toxic. However, both Na-OPP Fe-OPP are non-toxic and inexpensive, hence blending it with plant extract nanoparticles would produce cost effective and environmentally benign formulation.

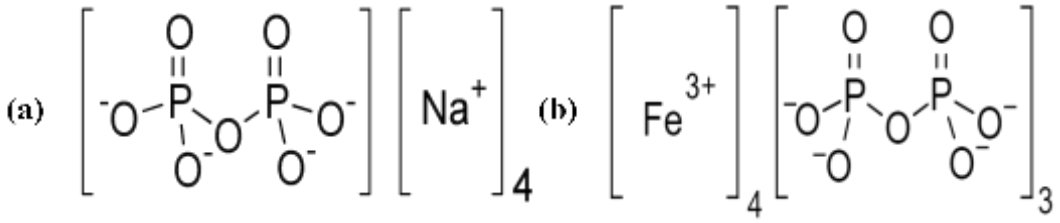

Figure 4. Molecular structure of (a) Na-OPP and (b) Fe-OPP.

The result of the blending (**Table 3**) revealed that the efficiency improved to 74.1 % for CAHE-ZnNPs + Na-OPP and 91.8 % for CAHE-ZnNPs + Fe-OPP at 30°C and 65.3 % for CAHE-ZnNPs + Na-OPP and 82.8 % for CAHE-ZnNPs + Fe-OPP at 60 °C, respectively. This increase in efficiency indicates the synergistic effect of both additives. When both Na-OPP and Fe-OPP were blended with CAHE-ZnNPs, the efficiency further improved to 97.2 % and 91.1 % at 30 °C and 60 °C, respectively. However, even in the presence of the additives, the inhibition efficiency still decreased with increase in temperature which signifies that temperature has an important influence on the performance of the formulations.

Table 3. Scale inhibition efficiency (%) on blending 250 ppm CAHE-ZnNPs with Na-OPP and Fe-OPP.

T (°C)	CAHE-ZnNPs + Na-OPP	CAHE-ZnNPs + Fe-OPP	CAHE-ZnNPs + Na-OPP + Fe-OPP
30	74.1	91.8	97.2
40	72.0	89.4	96.1
50	68.1	86.0	93.8
60	65.3	82.8	91.1

4. Conclusion

Cola acumulata husk extract - zinc nanoparticles (CAHE-ZnNPs) were synthesized, characterized and assessed as scale inhibitor in oilfield scaling medium. Near spherical crystalline nanoparticles with sizes around 76 – 83 nm and surfaces rich in C=C, C=O and –OH functional groups were obtained. The nanoparticles were 60.40 % efficient in inhibiting calcium carbonate scale at 30 °C. Inhibition efficiency increased with increase in concentration of CAHE-ZnNPs but decreased with increase in system temperature. On blending CAHE-ZnNPs with sodium pyrophosphate (Na-OPP) and ferric pyrophosphate (Fe-OPP), the scale inhibition efficiency increased to 74.1 % and 91.8 % at 30 °C but decreased to 65.3 % and 82.8 % at 60 °C respectively. When blended with both Na-OPP and Fe-OPP, the efficiency further increased to 97.2 % and 91.1% at 30 °C and 60 °C, respectively. The formulations of CAHE-ZnNPs with the additives exhibit high efficiency capable of being deployed in the industry. Therefore, instead of discarding *Cola acumulata* husks as waste, they could be converted into oilfield chemicals.

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