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Solvent-free Mechanochemical Synthesis, Characterization and Antibacterial Potency of $CaO@SiO_2$ Nanocomposite

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Authors' contributions

This work was carried out in collaboration among all authors. Authors DWL and ZS designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors MY and YDM managed the analyses of the study. Author SIA managed literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Objectives: This aim of this study was to synthesized $CaO@SiO_2$ nanocomposite using solvent-free mechanochemical method and applied against gram-positive bacteria (*Bacillus subtilis*, *Klebsiella pneunoniae*) and gram-negative bacteria (*Pseudomonas aeruginasa, Escherichia coli*, and *Salmonella typhi*).
Methods: The structure of $CaO@SiO_2$ nanocomposite was confirmed by FTIR and UV-Visible spectroscopy. Agar well diffusion method was used for the antibacterial studies. The antibacterial activity of $CaO@SiO_2$ nanocomposite was found to be dose dependent. The zone of inhibition decreases in the following order; *S. typhi*, *P. aeruginosa*, *K. pneumoniae*, *B. subtilis* and *E. coli*.
Conclusions: $CaO@SiO_2$ nanocomposite showed excellent antibacterial activity and can be used as promising antibacterial agent.

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Keywords: CaO@SiO_2 Nanocomposite; antibacterial; mechanochemical synthesis; solvent-free; environmentally friendly.

1. INTRODUCTION

Mechanochemical reactions occur between solids with no, or minimal, addition of solvents. Mechanochemical synthesis not only severely eliminates solvent waste, it also allows for quantitative and fast reactions to occur between the solids. In a milling synthesis, reagents are only exposed to one another at the colliding surface. The contact point of the reagents allows for product to be formed which breaks off of the original reagent particles and exposes more starting reagent for further reaction with continued milling. In mechanochemical synthesis, reaction set-ups occur in ball mills, such as shaker or planetary mills, with one or more balls of various sizes placed in together with the reagents. These could also be achieved simply using mortar and pestle [1-5]. Recently, some metal oxides, nanocomposites as well as metal organic framework were synthesized by mechanochemical methods. Thus, CuO/ZnO nanocomposite [1], ZnO nanoparticles [2], $Fe_3Al_2O_3$ nanocomposite [3], and Fe_3Al-Al_2O_3 nanocomposite [4] were synthesized using mortar and pestle. Also, copper isonicotinate metal organic framework was previously synthesized without addition of solvent by grinding the precursors with aid of mortar and pestle [5]. Therefore, different compounds could be synthesized using this simplest, fast and environmentally friendly method.

Clinical microbial strains have continued to develop adaptability or resistance towards antimicrobial drug/agents and this demands development of highly effective compounds for the treatment of critical microbial infection. Interesting, inorganic metal oxide nanoparticles such as CaO, ZnO, MgO, TiO_2, SiO_2 etc., are known to have antimicrobial properties and hence, it's widely applied as antimicrobial agents [6-16]. This aim of this study was to synthesize CaO@SiO_2 nanocomposite using solvent-free mechanochemical method and applied against gram-positive bacteria (*Bacillus subtilis*, *Klebsiella pneunoniae*) and gram-negative bacteria (*Pseudomonas aeruginasa, Escherichia coli*, and *Salmonella typhi*).

2. MATERIALS AND METHODS

2.1 Apparatus/Instruments

All the glasswares and apparatus used in this study are pyrex products. For this research the following apparatus were used; motar and pestle, siever, beakers, conical flasks, test tubes, measuring cylinder, whatman filter paper, watch glass, oven, weighing balance, funnels, hot plate, autoclave, petri dish, incubator, refrigerator, laminar flow cabinet, cotton wool, aluminium foil paper, candle, matches, universal container, cork borer, metre rule, masking tape, spatula, glass rod stirrer, syringes, micropipette, wire loop, Ultraviolet –Visible spectrophotometer (modelJENWAY 630), SEM, and FTIR (PerkinElmee Spectrum Version 10.0309).

2.2 Reagents

All the reagents used are BDH products.The reagents used in this research includes; Silica gel, $Ca(OH)_2$, Ethanol, Hydrochloric acid (HCl), Tetraoxosulphate (VI) acid (H_2SO_4), barium chloride (Bacl2), Normal saline, nutrient Agar, and Muller Hinton agar.

2.3 Synthesis of CaO@SiO_2 Nanocomposite

Solvent-free mechanochemical synthesis method was used in synthesizing CaO@SiO_2 nanocomposite in accordance as reported in the literature [1-5]. CaO@SiO_2 nanocomposite was prepared according to 1:1 ratio of calcium hydroxide as a precursor of CaO to silica gel as a precursor of SiO_2. The appropriate ratio of the precursors was mixed and grounded using mortar and pestle for one hour to obtain homogeneous mixture. The homogeneous mixture was calcined at 500ºC in furnace for 2 hours in order to get rid of water molecules associated with the precursors. Finally, the product was removed from furnace, cooled and stored for further analysis.

2.4 Characterization

The synthesized CaO@SiO_2 nanocomposite was characterized using spectroscopic techniques; Fourier Transform Infra-red (FTIR) and Ultraviolet-Visible spectroscopy.

2.5 Antimicrobial Activity Assay

The antimicrobial activity was tested by using agar-well diffusion method as described by Shehu et al. [17]. The bacterial isolates were first grown in a nutrient broth for 12–18 h before

use and standardized to 0.5 McFarland standards (10^6 cfu ml^{-1}). One hundred microliter of the standardized cell suspensions were spread on a Mueller-Hinton agar (Hi Media) and the agar medium was punched with a 6 mm diameter wells and filled with different concentration(100, 200, 300, 400 and 500 µg/L) of $CaO@SiO_2$ nanocomposite solutions in equal amounts. The plates are observed for zone of inhibition after 24 h incubation at 37ºC.

3. RESULTS AND DISCUSSION

3.1 UV/Visible Spectroscopy

The UV/Visible spectrum of $CaO@SiO_2$ Nanocomposite and $Ca(OH)_2$ are shown in Fig.1 whereas for the silica gel is shown in (Fig. 2). The maximum adsorption wavelength for $Ca(OH)_2$ and silica gel are 500 nm and 400 nm respectively. The surface plasmon resonance for the $CaO@SiO_2$ nanocomposite was found at 300 nm and it indicate the formation of nanocomposite (Fig. 1) [18].

3.2 Fourier Transform Infra Red (FTIR) Spectroscopy

The functional groups analyses were carried out using FTIR spectrometer. The FTIR of the precursor, silica gel is shown in (Fig. 3). The peaks observed includes; 3809.65 – 3471.85, 1643.19, 1083.71, 961.24, 788.11, 456.07, and 462.05 cm^{-1}. The band at 1083.71 cm^{-1} corresponds to assymetric stretching vibration of Si-O-Si bond. The peaks at 961.24 and 788.11 cm^{-1} corresponds to Si-OH bond. The peaks at 3809.65 – 3471.85 and 1643.19 cm^{-1}indicates H-O-H stretching and bending of adsorbed water. The peaks at 456.07 and 462.05 cm^{-1} correspond to Si-O bond. These reports are in agreement with previous report [13-16]. The FTIR spectra of the second precursor, Fig. 4, $Ca(OH)_2$ shows bands at 3644.98 cm^{-1}, 3437.40 cm^{-1}, 1636.40 cm^{-1}, 1428.00 cm^{-1}, 1125.47 cm^{-1}, 874.74 cm^{-1}, 466.80 and 455.11 cm^{-1}. The peaks at 3437.40 and 1635.07 cm^{-1}indicates H-O-H stretching and bending of adsorbed water. The sharp peak at 3642 cm^{-1} which is the characteristic band of $Ca(OH)_2$ is belong to the stretching mode of surface hydroxyl group. The peaks at 1473 cm^{-1} and 874.74 cm^{-1} correspond to the out of plane bending of CO_3^{2-} in $CaCO_3$. The observed bands at 455.11 and 466.68 cm^{-1} are relegated to metal oxide vibration peak for Ca–O stretching.

The FTIR spectra of $CaO@SiO_2$ nanocomposite, (Fig. 5) shows similar as in the precursors, (Fig. 3 and 4). In this case, the strong band at 3647 cm^{-1} corresponds to the O–H bonds from the remaining hydroxide, $Ca(OH)_2$ .The broad band around 1470.52 cm^{-1}, as well as a band at 87.87 cm^{-1} indicates the C–O bond related to carbonation of CaO nanoparticles [7-10,16]. The strong band at 455.00 cm^{-1} identified vibration of the Ca–O and Si-O bond [16]. The band at 1088.00 cm^{-1} corresponds to assymetric stretching vibration of Si-O-Si bond. The peaks at 874.87 and 792.00 cm^{-1} corresponds to Si-OH bond. The peak at 34451.52 indicates H-O-H stretching of silanol and adsorbed water [13,16]. These confirmed the formation of $CaO@SiO_2$ nanocomposite.

3.3 Antimicrobial Activity Results

Results for antibacterial studies of $Ca@SiO_2$ nanocomposite against *Bacillus subtilis*, *Klebsiella pneunoniae*,*Pseudomonas aeruginasa, Escherichia coli*, and *Salmonella typhi*) are presented in (Table 1 and Fig. 6).

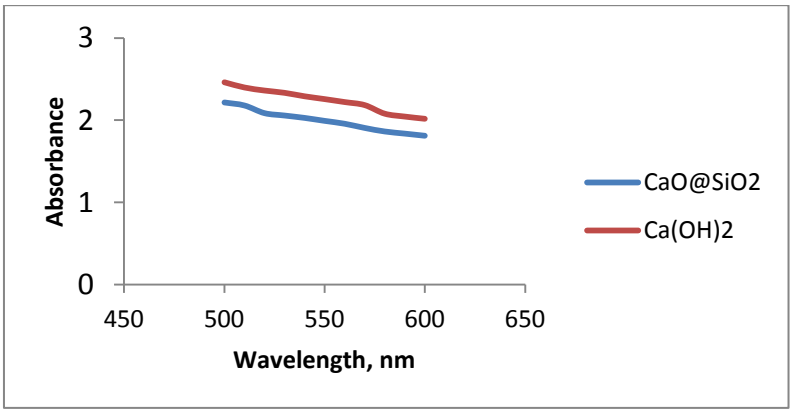

Fig. 1. Uv-visible spectrum for $Ca(OH)_2$ and $CaO@SiO_2$

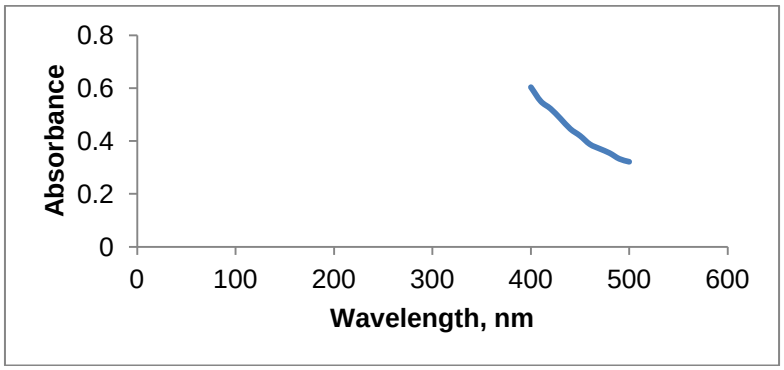

Fig. 2. Uv-visible spectrum for silica gel

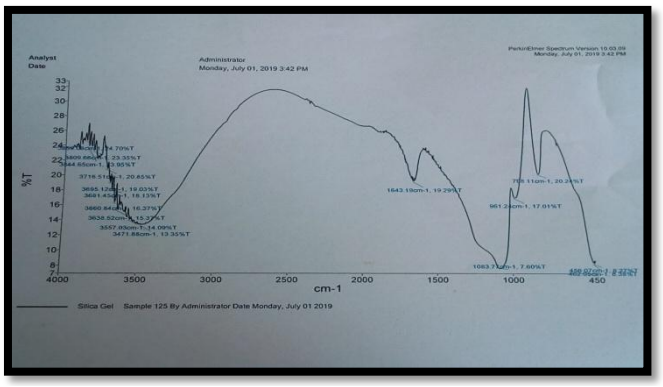

Fig. 3. Spectrum for silica gel

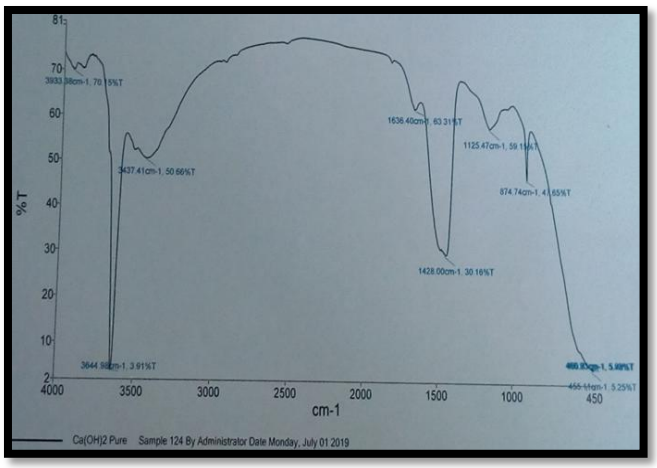

Fig. 4. FTIR spectrum of $Ca(OH)_2$

Augmentin was used as control throughout the studies at concentration of 300 µg/L. Different concentrations of 200, 300, 400 and 500 µg/L of $Ca@SiO_2$ nanocomposite was tested against each pathogen. Generally, the inhibition zone increases with increase in concentrations of $CaO@SiO_2$ nanocomposite for all the bacteria. Similar observations were report by Shehu et al. [17] and Shehu et al. [19]. At higher concentration of 500 µg/L, the zones of inhibition were in the following order; 26, 22, 20, 16, and 14 mm for S*. typhi*, *P. aeruginosa*, *K. pneumoniae*, *B. subtilis* and *E. coli* respectively. The zone of inhibition for control (Augmentin) was found to be higher compared to $CaO@SiO_2$ nanocomposite for each pathogen except for *P.*

aeruginosa. Augmentin and $CaO@SiO_2$ nanocomposite were found to have similar zone of inhibition of 22 mm for *P. aeruginosa* and this indicate effective of $CaO@SiO_2$ nanocomposite against the pathogen.Similarly, CaO nanoparticle was found to be effective against *P. aeruginosa* [6]. For each concentration investigated, *S. typhi* shows higher zone of inhibition as compared to other pathogens, Table 1 and Fig. 6. This shows that $CaO@SiO_2$ nanocomposite is more effective against *S. typhi* than any other pathogen under investigation for this study.

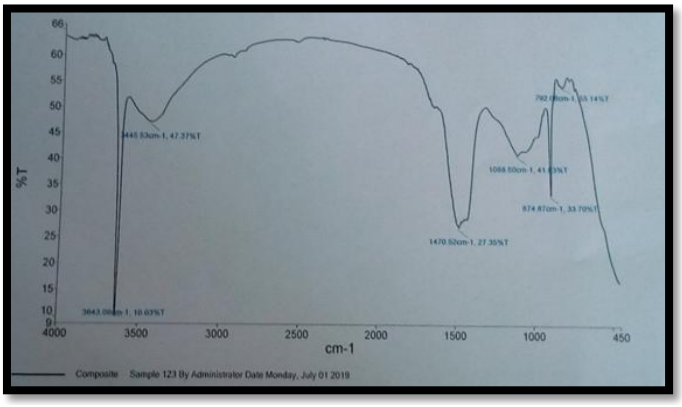

Fig. 5. FTIR spectrum of $CaO@SiO_2$ nanocomposite

Table 1. Zone of inhibition (mm) for $Ca@SiO_2$ nanocomposite against five bacteria pathogens

Concentrations of $Ca@SiO_2$ (µg/L)	*E. coli*	*B. subtilis*	*P. aeruginosa*	*K. pneumonia*	*S. typhi*
200	6	5	13	9	16
300	6	13	14	9	19
400	6	14	16	15	21
500	14	16	22	20	26
300 µg/L,Augmentin (control)	25	26	22	27	30

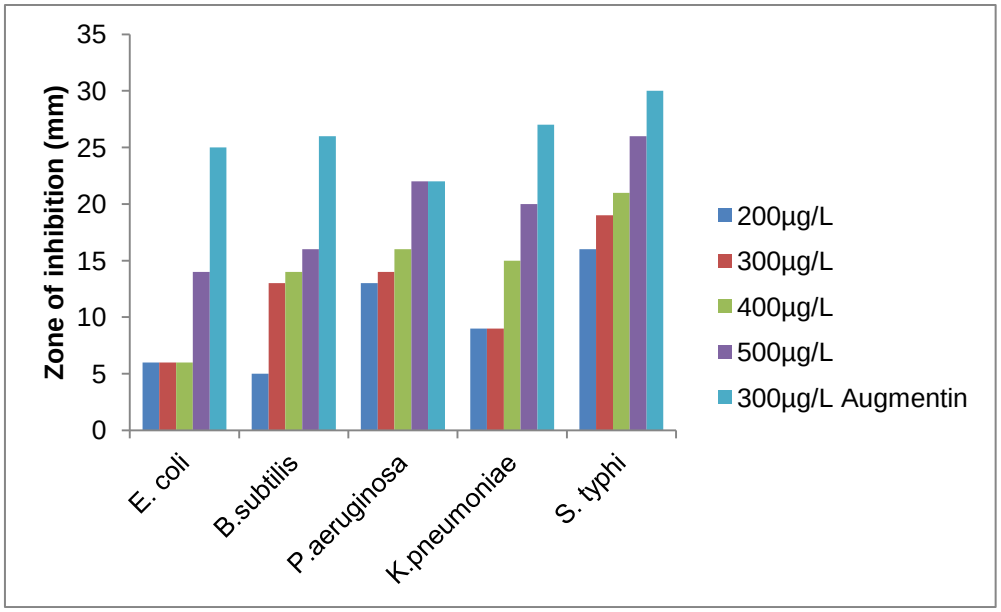

Fig. 6. Antibacterial activity of $CaO@SiO_2$ nanocomposite and control (Augmentin) against *E. coli, B. subtilis, P. aeruginosa, K. pneumoniae, and S. typhi*

4. CONCLUSION

Solvent-free mechanochemical method with aid of mortar and pestle was used in synthesizing $CaO@SiO_2$ nanocomposite. The structure of $CaO@SiO_2$ nanocomposite was confirmed by FTIR and UV-Visible spectroscopy. The zones of inhibition for for *S. typhi*, *P. aeruginosa*, *K. pneumoniae*, *B. subtilis* and *E. coli* showed excellent antibacterial activity and can be used as promising antibacterial agent.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

1. Patil GG, Sayyad SB. Study the Synthesis of CuO/ZnO nanocomposite by Mortar and Pestle and its Characterization. Journal of Emerging Technologies and Innovative Research. 2017;4(11):543-546.
2. Florence SS, Adam H, Manna C, Can N. Green approach to synthesis and strain studies of ZnO nanoparticles, AIP Conference Proceedings. 1976, 020020. 2018;1-4.
3. Khodaei M. Kinetics of Fe_2O_4-Alreaction prior to mechanochemical synthesis of $Fe_3Al_2O_3$ nanocomposite powder using thermal analysis, J. Nanoanalysis. 2019; 6(3):199-204.
4. Khodaei M, Enayti MH, Karimzadeh M. Mechanically synthesized Fe_3Al-Al_2O_3 nanocomposite, J. Alloys and Comp. 2009; 467:159-162.
5. Tella AC, Owalude SO, Ojekanmi CA, Oluwafemi OS. Synthesis of copper isonicotinate metal-organic frameworks simply by mixing solid reactants and investigation of its adsorptive properties in the removal of fluorescein dye. New Journal of Chemistry. 2014;38:4494–4500.
6. Arup R, Samiran SG, Madhusmita B, Jayanta B. Antimicrobial Activity of CaO Nanoparticles, Journal of Biomedical Nanotechnology. 2013;9:1–8.
7. Karthik K, Dhanuskodi S, Gobinath C, Prabukumar S, Sivaramakrishnan S. Dielectric and antibacterial studies of microwave assisted calcium hydroxide nanoparticles, J Mater Sci: Mater Electron. 2017;1-11.
8. Doaa MAE, Naeima MHY. Antimicrobial Effects of Calcium Oxide Nanoparticles and Some Spices in Minced Meat. ARC Journal of Animal and Veterinary Sciences. 2017;3(2):38-45.
9. Risfidian M, Kiki A, Fahma R, Aldes L. Preparation Calcium Oxide (CaO) from Chicken Eggshells, SJE. 2016;1(2):32-35.
10. Bakhtiari F, Darezereshki E, Esmaeilzadeh E. Preparation and characterization of CaO nanoparticles from Ca(OH)2 by direct thermal decomposition method, Journal of Industrial and Engineering Chemistry. 2014;20:113–117.
11. Joshi NC, Ramola B, Ramola M, Chhabra J, Singh A. Green Synthesis, Characterisations and Antimicrobial Activities of CaO Nanoparticles, Orient. J. Chem. 2019;35(3):1154-1157.
12. Ashwini A, Ramalakshmi S, Mary G. Green Synthesis of Calcium Oxide Nanoparticles and Its Applications, Int. Journal of Engineering Research and Application. 2016;6(10) part 1:27-31.
13. Irvan D, Gui MM, Azlina HK, Abdul RM, Keat TL. Removal of SO_2 and no over Rice Husk Ash (RHA)/Cao-supported metal oxides, Journal of Engineering Science and Technology. 2008;3(2):109–116.
14. Alfred AC. Effect of Heat on the Adsorption Properties of Silica Gel, *IACSIT* International Journal of Engineering and Technology. 2012;4(4):484-488.
15. Aya V, Arpita B. Analysis on Synthesis of Silica Nanoparticles and its Effect on Growth of *T. Harzianum* & *Rhizoctonia* Species. Biomed J Sci&Tech Res. 2018; 10(4):1-8.
16. Aymen H, Abdelali M, Ouanassa G, Mohamed G. Synthesis and characterization of bioactive tannery SiO_2-CaO-P_2O_5 Bioglass, International Journal of Engineering and Applied Sciences (IJEAS). 2018;5(2):23-28.
17. Shehu Z, Danbature WL, Maisanda AS, Musa MS. Synthesis, Characterization and Antibacterial Activity of Kaolin/Gum Arabic Nanocomposite on Escherichia Coli and Pseudomonas Aeruginosa, Research.

Journal of Nanoscience and Engineering. 2018;2(2):23-29.

18. Alfred AC. Effect of Heat on the Adsorption Properties of Silica Gel, IACSIT International Journal of Engineering and Technology. 2012;4(4):484-488.

19. Shehu Z, Lamayi DW, Sabo MA, Aliyu SI. Kaolin/Gum Arabic Nanocomposite as an Antifungal Agent Against *Aspergillus flavus* and *Saccharomyces cerevisiae*, Iraqi Journal of Science. 2019;60(11):2316-2320.

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