



Syntheses and characterization of *Syzygium aromaticum*, *Elettaria cardamomum* and *Cinnamomum verum* modified TiO₂ and their biological applications

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Abstract

Diseases form the major roadblock for the living organism. Many chemicals are isolated to cure diseases. But they are found to have side effects due to allergic chemicals. NPs are found to be good biological agents. But they are also allergic in nature. In this study, the properties of TiO₂ NPs were modified with bio agents such as Clove, Elachi (Cardamom) and Cinnamon. The anticancer and antibacterial properties were also examined. XRD analysis showed the anatase phase for the pure and modified TiO₂ samples. The particle sizes were found to be 7.5 nm for pure, 9.5 nm for Clove modified, 9 nm for Elachi modified and 10.5 nm for Cinnamon modified TiO₂ NPs. The synthesized TiO₂ nanoparticles were tested against bacterial strains such as *Escherichia coli*, *Klebsiella pneumoniae*, *Pseudomonas aeruginosa*, *Staphylococcus aureus* and *Streptococcus mutans* using agar diffusion method for antibacterial studies. KB Oral cancer cell line was used to determine the anticancer activities of the synthesized NPs by MTT assay. The synthesized bio modified NPs exhibit significant results against bacterial strains and cancer cell line when compared with the pure NPs.

Introduction

Nanomaterials are gaining more attraction in day to day life for more than decades. They exhibit significant applications in different fields like biology, chemistry, biotechnology and many more research areas due to their excellent properties [[1], [2], [3], [4]]. Antibacterial and anticancer activities of the NPs are mainly due to their Reactive Oxygen Species (ROS) [5]. Many nanomaterials such as Zn [6,7], Cu [8] and Ag [4] exhibit antibacterial and anticancer activities. Among them TiO₂ NPs showed excellent photocatalytic activity [9] and has a tendency to kill cancer cells [[10], [11], [12], [13]]. Jin C et al. [14] observed that the anatase phase of TiO₂ was able to generate more ROS than the rutile phase and was experimentally studied using XAFS (X-ray absorption fine structure spectrometry). The biological activities of TiO₂ NPs increases with the doped and modified chemical agents. But they are found to be more toxic and expensive. In order to reduce the toxic nature and cost, plant materials can be used as dopants. Plants are found abundant in the environment and they have been used from ancient time in many medicinal systems such as Siddha, Ayurvedha etc. to cure many diseases. From the literature, it is reviewed that spices have a tendency to destroy bacterial [15] and cancer cells [16,17]. Among them Clove, Cardamom and Cinnamon are found to exhibit excellent biological activities. Hence modified TiO₂ NPs have been prepared using Clove, Cardamom and Cinnamon to enhance the antibacterial and anticancer activities.

Cloves are the aromatic flower buds which belong to the Myrtaceae family and the botanical name is *Syzygium aromaticum*. Chaeib K et al. [18] studied the chemical composition of Clove (*Syzygium aromaticum*) and observed that eugenol (76.8%), β - caryophyllene (17.4%), α - humulene (2.1%), and eugenyl acetate (1.2%) are the main components. Kumar Y et al. [19] observed the antibacterial activity of Clove with two Gram positive (*Bacillus cereus*, *Staphylococcus aureus*) and two Gram negative (*Salmonella typhi*, *Escherichia coli*) bacterial strains for various concentrations at 2000, 1500 and 1000 ppm using 5 mm disc diameter. Liu H et al. [20] studied the anticancer activity of *Syzygium aromaticum* using HT - 29 Cells and observed that the ethyl acetate extract of Clove (EAEC) and Oleanolic acid (OA) present in Clove exhibits cytotoxicity against several human cancer cells.

Elachi (Cardamom) is a spice made from the seeds of genera plants *Elettaria*. The botanical name is *Elettaria cardamomum*. Korikanthimathm V S et al. [21] observed 1,8-Cineole (36.3%), alpha - teripinyl acetate (31.3%) and limonene (11.6%) to be the major constituents of the Cardamom. The basic aroma produced by Cardamom is mainly due to these components. The other components present in cardamom are pinene, sabinene, myrcene, phellandrene, terpinene, cymene, terpinolene, linalool, linalyl acetate, terpineol, citronellol, nerol, geraniol, methyl eugenol and nerlidol. Akrayi H F S [22] studied the antimicrobial activity of *Elettaria cardamomum* against *Staphylococcus aureus* and *Proteus mirabilis*. Majdalawieh Amin F et al. [23] investigated the anticancer and antitumor activity of Cardamom and observed that the aqueous extracts of Cardamom significantly increase the destruction of cancer cells and found that they are natural killer cells.

The botanical name of Cinnamon is *Cinnamomum verum*. It is found in the inner bark of the tree. Meena V [24] pointed out that the major constituents of Cinnamon bark are cinnamaldehyde (65–80%) and eugenol (5–10%). The component Cinnamaldehyde is responsible for the pungent taste and odour. The other compounds present in traces are cinnamic acid, hydroxyl cinnamaldehyde, cinnamyl alcohol, coumarin, cinnamyl acetate, borneol, β -caryophyllene, benzyl benzoate, linalool and eugenyl acetate.

Ali N A M et al. [25] studied the chemical composition and antimicrobial activities of Cinnamon bark and leaf extracts and concluded that the Cinnamon bark has efficient antibacterial activity than leaf extracts due to their major components present in the bark. Wang H M et al. [26] showed the cytotoxic nature of Cinnamon against human skin cancer melanoma A375.S2 cells. Fig. 1 represents the chemical structure of eugenol, oleanolic acid, 1,8-cineole and cinnamaldehyde.

Titanium dioxide NPs are synthesized by various methods such as sol gel [27], coprecipitation [28], hydrothermal [29,30], spray pyrolysis [31], DC reactive magnetron sputtering [32]. Tam K H et al. [33] suggested that hydrothermal methods are gaining more interest because of their low cost and environmentally friendly method.

In the present work, Pure, Clove modified, Elachi modified and Cinnamon modified TiO₂ were synthesized by hydrothermal technique. They were subjected to characterizations such as XRD (X-Ray diffraction), UV-vis (UV-vis Spectrophotometer), FTIR (Fourier Transform InfraRed spectrometer), TEM (Transmission Electron Microscopy), MTT assay and Agar diffusion technique. The modified samples exhibit better activity than the unmodified samples.

Section snippets

Experimental methods

Titanium tetraisopropoxide and isopropyl alcohol were purchased from Sigma - Aldrich Chemicals. Dried Clove buds, Cardamom and Cinnamon were purchased from local Supermarket....

X-ray diffraction analyses

Fig. 2 **depicted** the X-ray patterns for **T1, T2, T3, T4, T5, T6 and T7** NPs. The peaks observed at 25.3°, 37.8°, 48°, 54.7°, 63°, 70° and 75.7° denotes the crystal planes of (1 0 1), (0 0 4), (2 0 0), (1 0 5), (2 0 4), (2 2 0) and (2 1 5), respectively, which is in good agreement with the JCPDS file No: 21-1272 of anatase TiO₂ [34]. Nainani R et al. [35] have synthesized silver doped titanium dioxide NPs by photodeposition technique and the size of the synthesized particle was found to be 7 nm....

Conclusion


The *Syzygium aromaticum*, *Elettaria cardamomum* and *Cinnamomum verum* modified NPs were synthesized using hydrothermal method and characterized using XRD, UV, FTIR and TEM analysis. The research experiments analysed the biological activities of the synthesized samples which confirmed that the modifiers have enhanced the biological activities of the TiO₂ NPs. The inhibition bacterial zones are found to be maximum for *Syzygium aromaticum* modified TiO₂ samples in comparison with the pure samples...

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