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RESEARCH PAPER

Investigation of Capecitabine Loading on Graphene Oxide/Magnesium Oxide/Chitosan Nanostructures for Drug Delivery Applications

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ABSTRACT

In recent years, nanocarriers composed of polymers, liposomes, and other materials have gained attention for the delivery of nucleotides and therapeutic drugs. This study investigates the use of graphene oxide (GO) as a key component in nanocarriers, owing to its unique chemical structure, high surface area, biocompatibility, and versatile functional properties. GO exhibits strong potential for applications in drug and gene delivery, tissue engineering, and other biomedical fields due to its ability to interact effectively with various compounds. In this work, a novel GO–MgO–chitosan nanocarrier was synthesized and evaluated for drug loading and release capabilities. Comprehensive characterization techniques—including Fourier-transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), X-ray diffraction (XRD), Raman spectroscopy, thermogravimetric analysis (TGA), and zeta potential analysis—were conducted before and after drug incorporation. These analyses confirmed the successful fabrication of the nanocarrier and provided insight into structural, morphological, and thermal changes associated with drug loading, supporting its potential in targeted drug delivery systems.

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INTRODUCTION

Due to facilitating good targeting, controlled drug release, and efficient loading, nanoscale drug delivery systems such as nanospheres, nanocapsules, micelles, carbon nanotubes, nanosheets, nanotubes, and nanoliposomes have gained considerable attention at the interface of biomedical applications and nanotechnology in recent times [1-3]. Graphene (GO) is a two dimensional crystal and thin layer of carbon atoms that are Graphene is a thin layer of carbon atoms. It is a two-dimensional crystal that is connected in a hexagonal network that resembles a honeycomb. Because of its unique properties, such as its large surface area, high electromagnetic and conductive properties, and its chemical and physical properties, it has gained great interest and is used in wide applications [4]. Due to the thin atomic layer of graphene, its chemical stability and high mobility of carriers at room temperature, it has an important role in preparing nanocomposites [5]. Graphene is also considered

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to have high thermal conductivity, is wide-ranging, low-cost and is considered a semiconductor with a zero gap [6]. Magnesium oxide [MgO] It is a basic alkaline earth metal oxide with key advantages such as thermal stability, high pH, low toxicity, low cost, a high surface hydroxyl group, and a large surface area [7,8]. Due to its properties of low electrical conductivity, high thermal stability, and catalytic behavior, magnesium oxide is used as a therapeutic agent in the pharmaceutical and paint industries, toxic waste treatment, and superconductors. Magnesium oxide particles are also prepared by hydrothermal, sol-Gol, laser evaporation, wet chemical methods, and other methods. All of these methods produce high-cost, highly toxic magnesium oxide nanoparticles. This nanoparticle also has wide interest in medical and biological applications. Therefore, it is used as a biocide, bone regeneration, cancer treatment, and heartburn treatment. Therefore, green synthesis is an alternative method for producing non-toxic magnesium oxide for medical and biological applications.[9]. Capecitabine (cap) It has the chemical name pentyl [1-(3,4-dihydroxy5-methyl-tetrahydrofuran-2-yl)-5-fluoro-2-oxo-1Hpyrimidin-4-yl] amino methanoate, as shown in Fig. 1 It is an antitumor chemotherapy drug used in the treatment of metastatic rectal, colon and breast cancer. It is taken orally. Its molecular formula of $C_{15}H_{22}FN_3O_6$ and molecular weight of 359.3 have

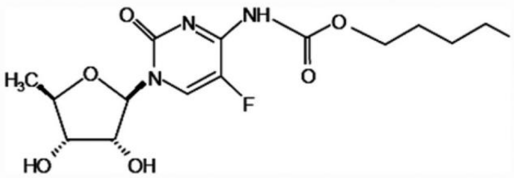

Fig. 1. Structure of capecitabine.

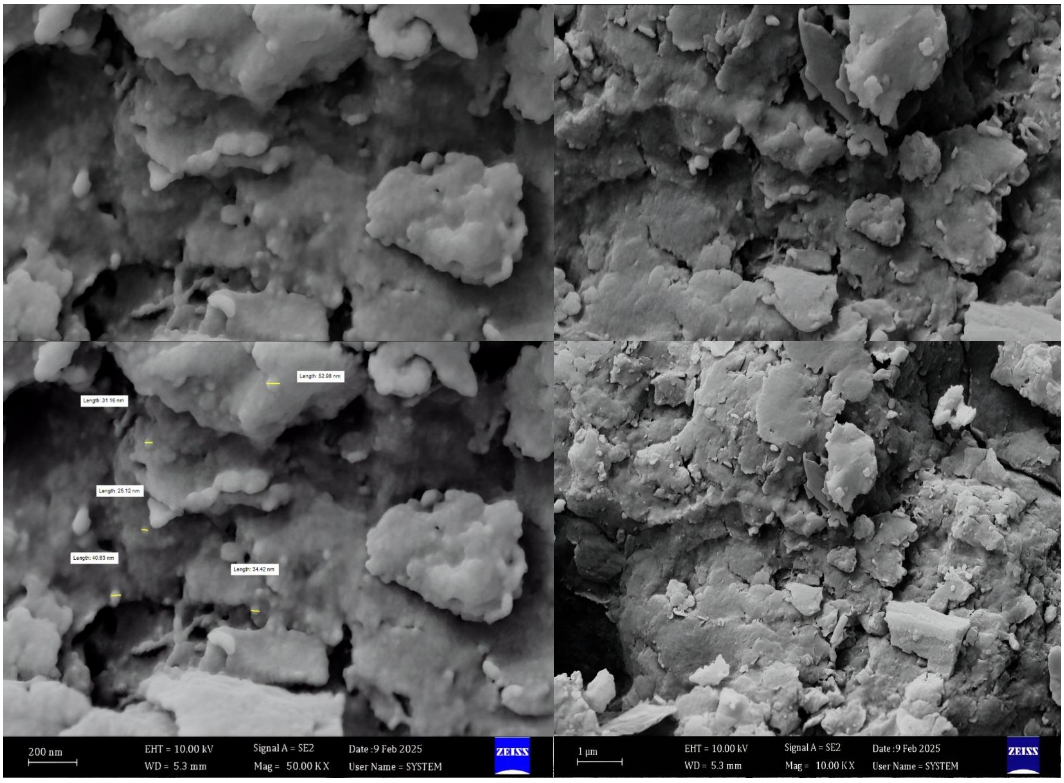

Fig. 2. SEM of GO-MgO-chitosan.

a short half-life and are in the form of a white powder.[10-12]. Chitosan, It is considered a family of linear polysaccharides and copolymer composed of glucosamine and N-acetyl glucosamine derived from chitin.It is soluble in acidic aqueous media via the proton of the primary amine.Due to its amino and hydroxyl functional groups, chitosan has wide-ranging applications in medicine, food, industry, agriculture, and more.which is a major component of crustacean shells and one of the most abundant biopolymers in nature [13-15].

MATERIALS AND METHODS

Materials

capecitabine was obtained from med chem express (MCE) amrica chitosan was purchased

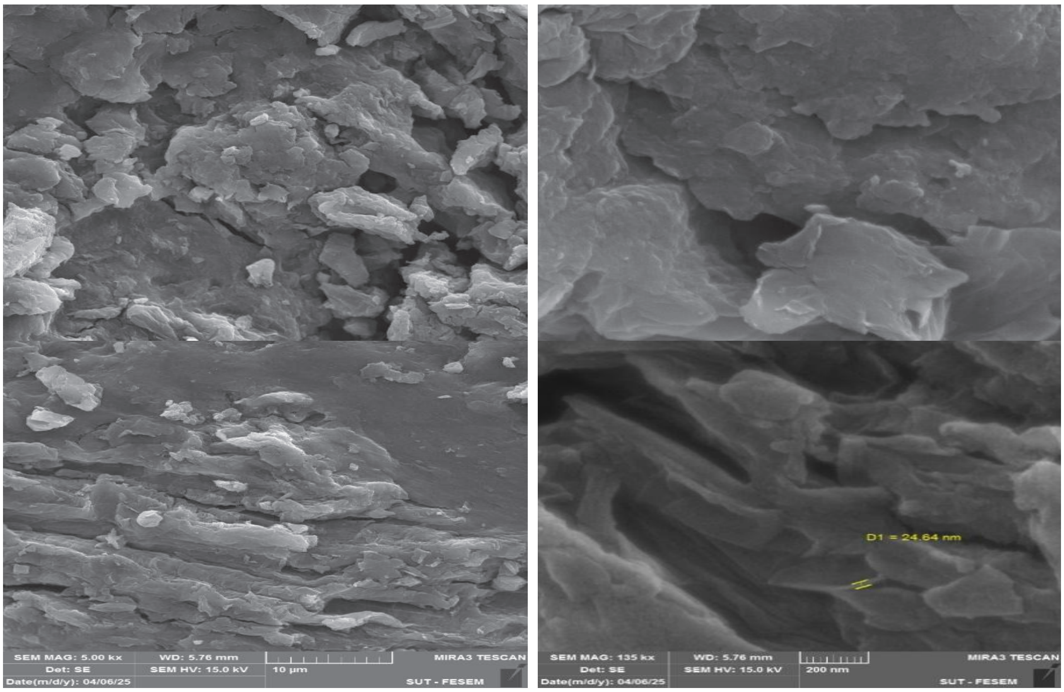

Fig. 3. SEM of GO-MgO-chitosan- cap.

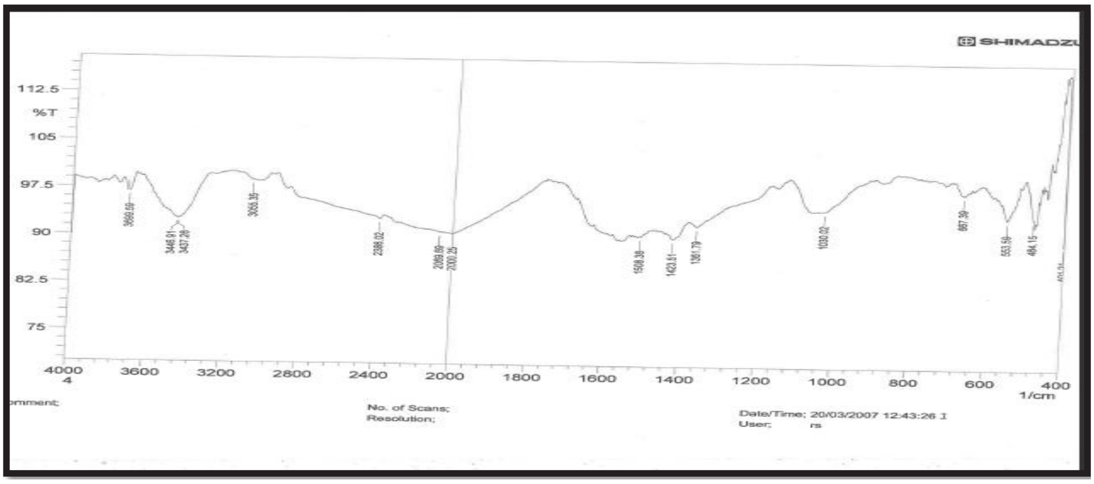

Fig. 4. FT-IR of GO-MgO-chitosan.

from india phosphate-buffered saline (PBS) were purchased from Hi Media (India). Glycine was purchased from Media (India). dialysis membranes were purchased from USA. The pH meter from OAICTON 2100. water.

Synthesis of GO and GO-MgO

Graphene oxide (GO) powder were prapeard following the modified Hummer method. Two grams of graphite were mixed with 100 ml of concentrated sulfuric acid at room temperature and stirred for half an hour. 12 g of potassium permanganate was slowly added to the mixture. The solution was stirred for one hour in an ice bath, then left to stand for another hour in an ice bath. The solution was then stirred for four hours in a water bath at 50°C. 200 ml of deionized water was then added to the mixture, which turned a yellow-brown color. 400 ml of water was then slowly added to the mixture and allowed to stand for 10 minutes. 20 ml of hydrogen peroxide was then added to the mixture and left to stand for one hour at 100°C. The resulting mixture was then centrifuged, washed, and dried at 70-80°C. Synthesis of GO-MgO Powder. 40 mg of synthesized GO powder was dispersed in 100 ml DI water and playced sonication for 1h. It led to formation of a stable GO suspension. Next, 40 mg

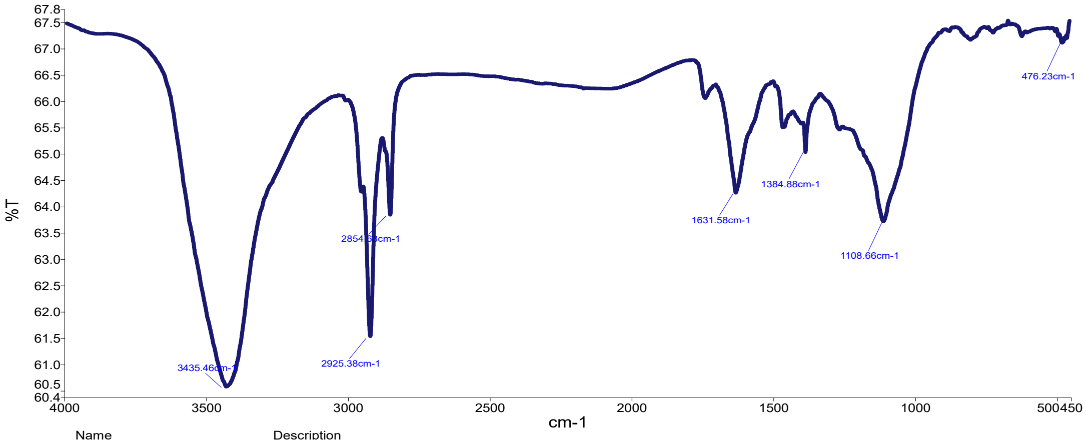

Fig. 5. FT-IR of GO-MgO-chitosan- cap.

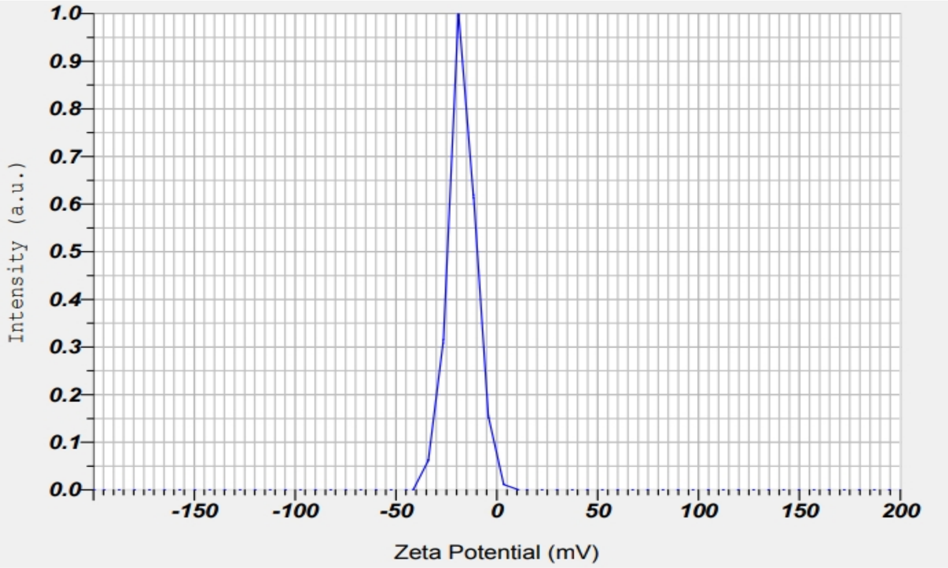

Fig. 6. Zeta potential of GO-MgO-chitosan.

of $Mg(NO_3)_2.6H_2O$ was dissolved in 100 ml DI water. In this solution was added to the GO suspension. The mixture was left for half an hour, then ultrasonicated for 4 hours. The resulting mixture was then centrifuged, filtered, and washed several times with deionized water, followed by ethanol three times. It was then air dried [16].

Synthesis of GO- MgO-chitosan- capecitabine

To synthesis GO- MgO-chitosan (0.25 g) chitosan mixed acetic acid (2%). Added 0.5% (w/v) mgCl2 and 0.5% (w/v) graphene oxide were kept in the constant bath stirring. Bath temperature was gradually elevated to 70ºC and fixed for 2 h. NaOH 45% (w/v) aqueous was blended gradually until a dark black precipitate formation. After 24h. the precipitate was clean and filter. The final GO- MgO- chitosan residue was dried at 100ºC for 2 h in a hot oven. the Loading of cap to GO-MgO- chitosan was carried out by stirring different amounts of cap (0.003 g to 0.005 g) overnight with GO- MgO- chitosan at a GO concentration of 0.02 g in ethanol. Unloaded cap was removed by

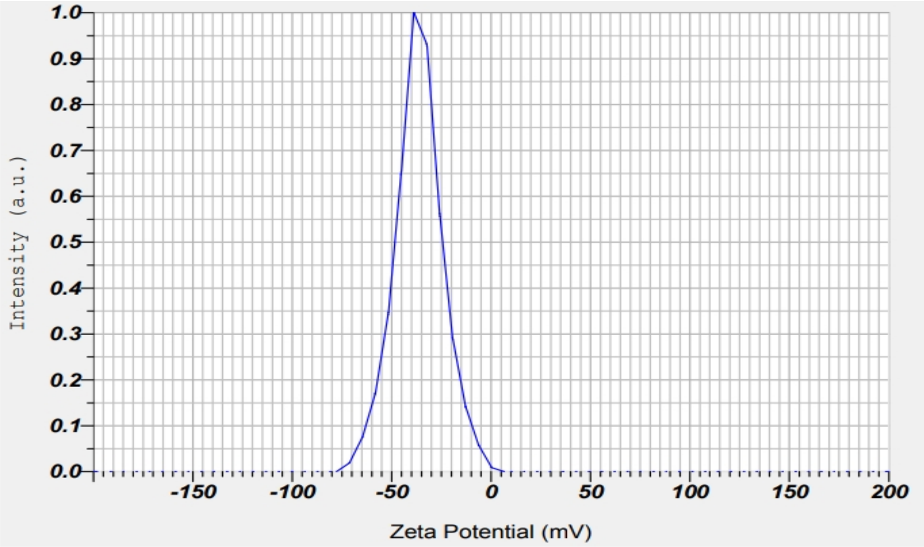

Fig. 7. Zeta potential of GO-MgO-chitosan-cap.

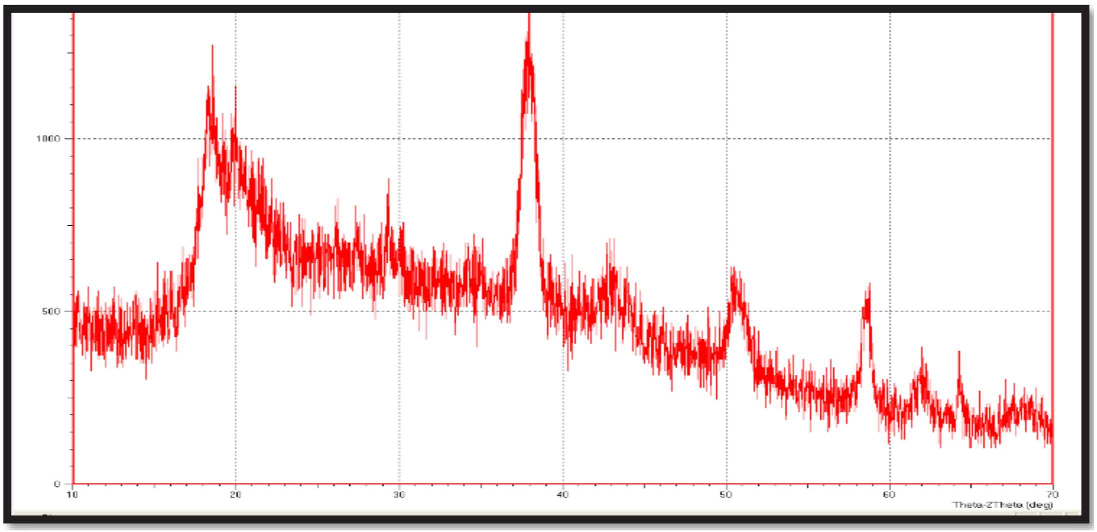

Fig. 8. XRD of GO-MgO-chitosan.

centrifugation for 5 min and its concentration was measured by UV-VIS-NIR spectrometer at (301 nm) The sample was washed five times with water [17,18].

Characterization

Fourier transform infrared measurements of GO- MgO- chitosan, and GO-MgO- chitosan -cap were carried out using a Bruker Vertex 80 IR spectrometer (Germany) at a resolution of 4 cm^{-1} from 4000-400 cm^{-1}. Raman spectroscopic characterization was done with Bruker Senterra (Germany) excited at 532 nm laser line. Ultraviolet-visible near infrared (UV-VIS-NIR) spectra were recorded using a UV-3600 (Shimadzu, UV-VIS-NIR, Japan). Thermal decomposition of were analyzed using a SDT Q600 thermo gravimetric analyzer (TA Instruments) from 25_C to 1000_C using a ramp rate of 10_C/min in air.

Determination of Encapsulation Efficiency, Loading Capacity

The amount of incorporated cap in GO-MgO- chitosan composite was determined

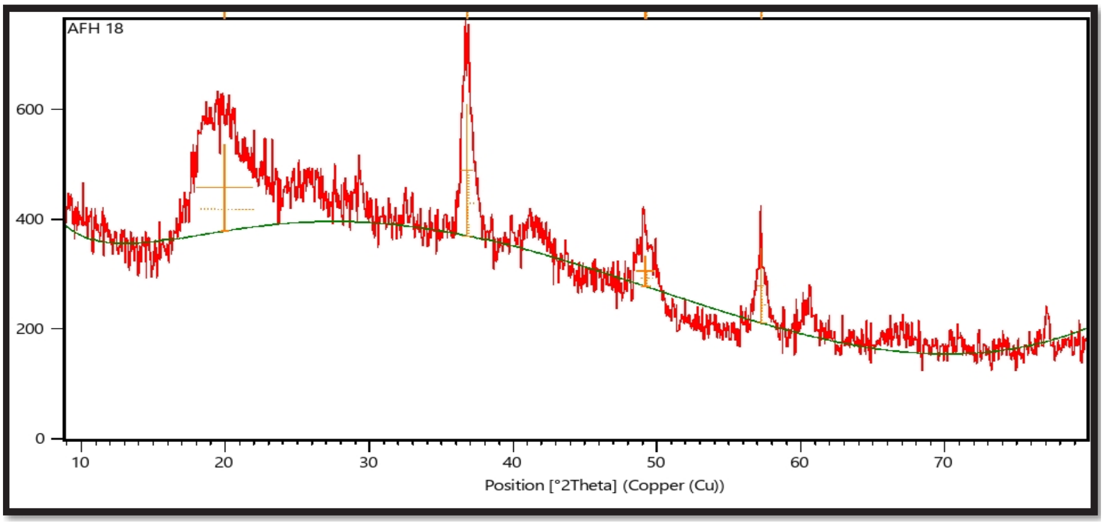

Fig. 9. XRD of GO-MgO-chitosan- cap.

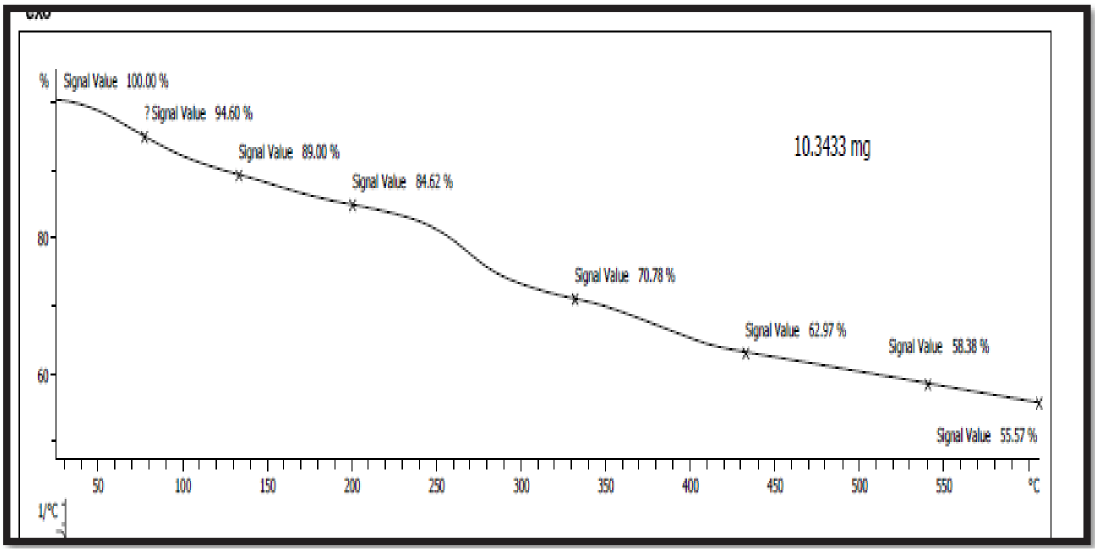

Fig. 10. TGA of GO-MgO-chitosan.

through in the supernatant remain after centrifugation of the reaction mixture, using the UV-visible spectrophotometer at 301 nm. Then the concentration was calculated from a calibration plot obtained for pure cap. Percentage encapsulation efficiency was calculated as follows.

cap Encapsulation (%)=([m cap−m U] / m cap)×100

m cap= total amount of cap added

m U=Remaining amount of drug in the supernatant

%Loading capacity = (m cap- m U) / m

m=final weight of the composite

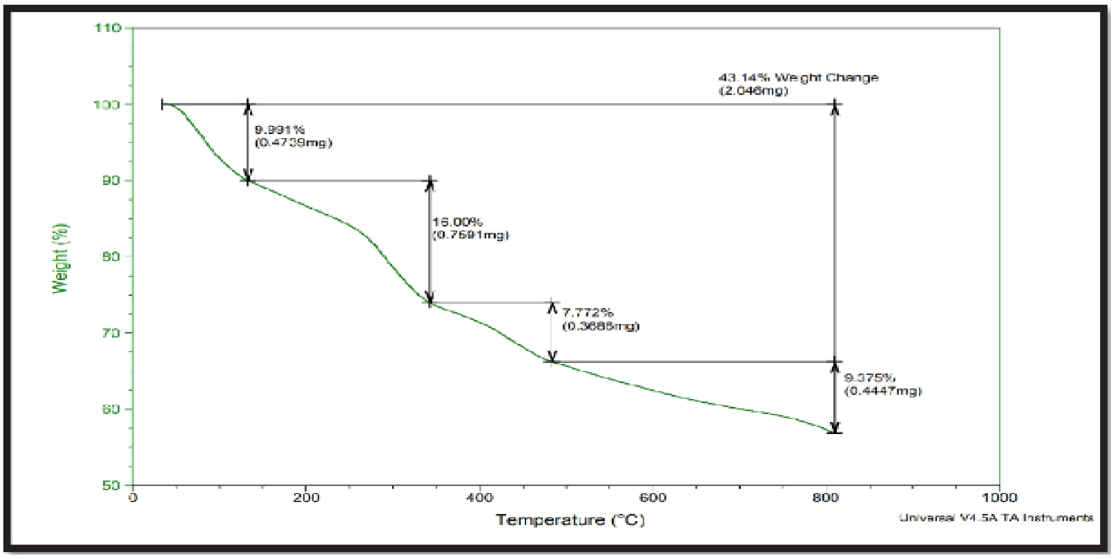

Fig. 11. TGA of GO-MgO-chitosan- cap.

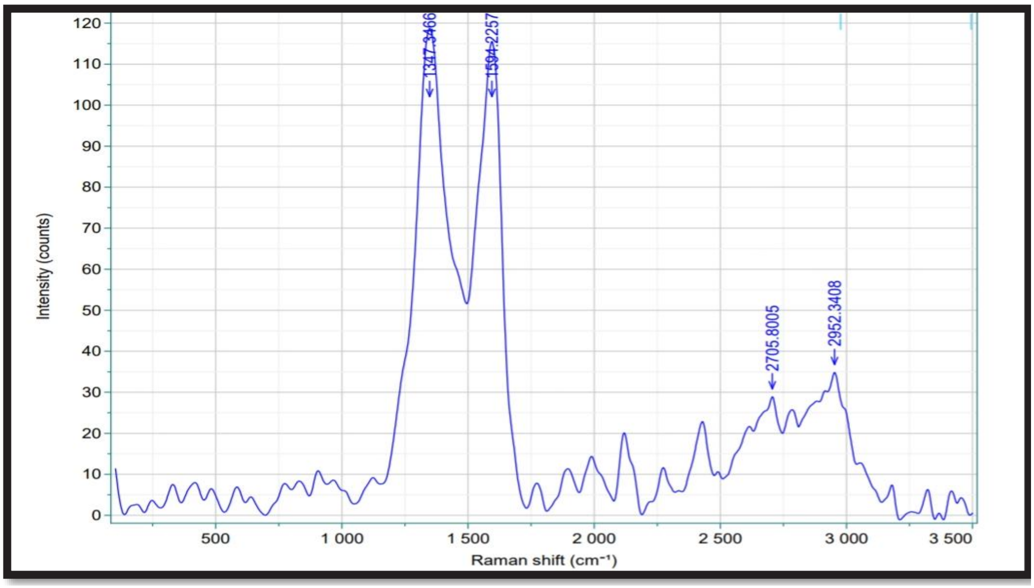

Fig. 12. Raman of GO-MgO-PEG.

Release of capecitabine

The release of cap from the GO- MgO- chitosan - cape composite were studied in three mediums phosphate-buffered saline (PBS) solution at neutral pH 7.4 and pH=8 and PH= 2.8 (Glycine Hcl buffer). The cap -loaded nanocomposite was dispersed in 5.00 mL of water and trapped inside a dialysis membrane and then immersed in 100mL of buffer solution at 25 C° with mild agitation. Aliquots (5 mL) were withdrawn at predetermined time intervals and their UV absorbance was measured at 301 nm using a UV-visible spectrophotometer

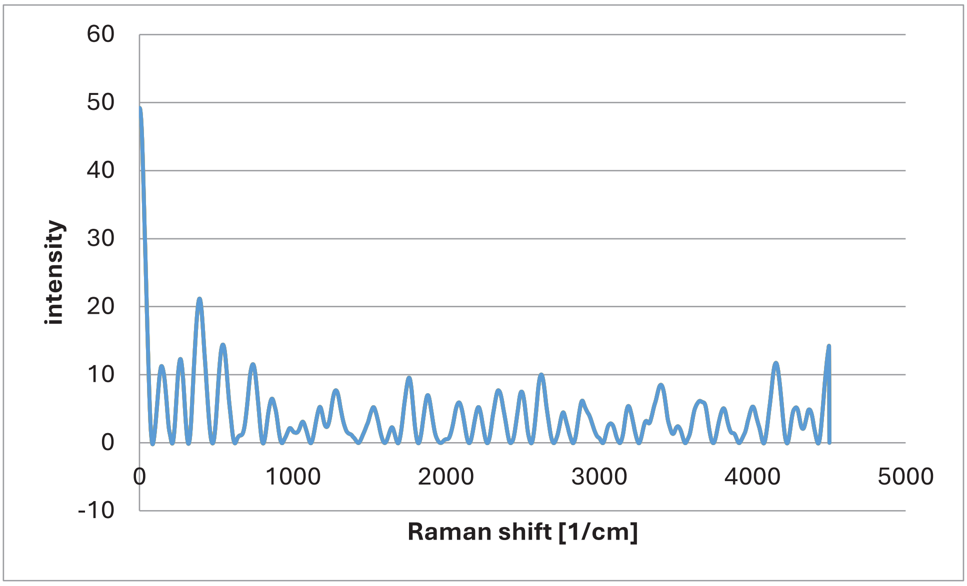

Fig. 13. Raman of GO-MgO-PEG-cap.

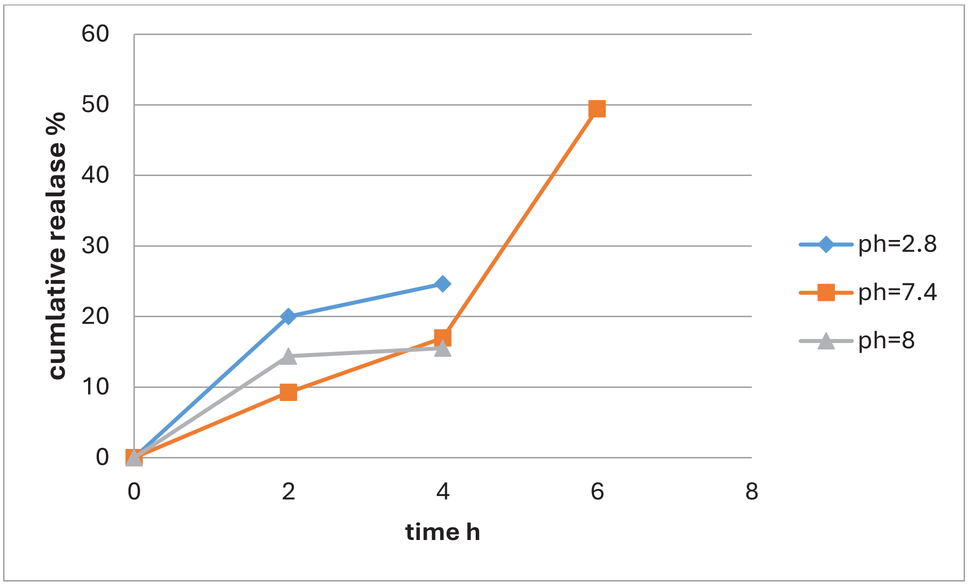

Fig. 14. Cap release for different Ph.

[19].

RESULTS AND DISCUSSION

Characterization of GO- MgO- chitosan - cap

The morphology of the nanostructure of GO-MgO- chitosan the compound was studied before and after cap loading it with the drug using a scanning electron microscope. The Fig. 2 shows the interpenetration of magnesium oxide and chitosan between the graphene oxide sheets in a coherent manner, and that the nanostructure is smooth and envelops the chitosan sheets within the graphene oxide sheets. Fig. 3 shows scanning electron microscope images after loading cap onto GO- MgO- chitosan the nanostructure. The appearance of slight differences and changes in the shapes after loading the drug onto the surface of the nanostructure is evidence of the success of loading the drug onto the surface of the compound GO- MgO- chitosan.[20]. Fig. 4 FTIR spectra exposes the O–H stretching band around ~3446.91 cm^{-1}. The C–H symmetric and asymmetric vibrations observe at ~2386.20 and ~2069.69 cm^{-1}. A C=C vibration exhibits nanocomposites at 1508.38 cm^{-1} due to GO presence respectively. the band at ~553.59-484.15 cm^{-1} implies the anti symmetric and symmetric vibrations of Mg-O recognized from MgO on GO-chitosan surface. In Fig. 5 the FT-IR spectra for GO-MgO-chitosan - capecitabine was explained, in comparison with GO-MgO-chitosan, GO-MgO-chitosan - capecitabine showed the most characteristic bands of each though with slight shifts at 3435.46cm^{-1}, 1108.66 and 1384 .88 cm^{-1}. These shifts may be attributed to changes in the molecular environment or interactions between the components, indicating the successful formation of GO- MgO- chitosan-cap nanocomposite [21].

Zeta potential for GO- MgO- chitosan and GO-MgO-chitosan after loading cap are -17.1 and -36.3, respectively as shown in the Fig. 6 and Fig. 7 which represents our nanocarrier has a highly negative surface charge. Therefore, it seems our compound has properly potential for using in drug delivery [22].

Fig. 8 shows the XRD pattern of GO- MgO-chitosan. In this figure, a sharp diffraction peak at 2h = 18.5, 29.5, 38.5, 51,58.5, 62 and 64.5 is detected, suggesting the formation of GO- MgO-chitosan After loading cap into the GO- MgO-chitosan nanocomposite, the XRD patterns Fig. 9 showed minimal change compared to the GO-MgO- chitosan composite, though boarder peaks were observed. [23].

TGA curve of GO- MgO- chitosan is shown in Fig. 10. The TGA curve represented weight loss of around 94.60 % in the sample at temperature of about 70°C and 89.00% at 130 °C and 84.62 at 200 °C and 70.78 % at 330 °C and 62,67 % at 430 °C and 58.38 % at 540 °C which indicates the thermal stability of the sample.TGA curve of GO- MgO-chitosan after loading cap is shown in Fig. 11 The TGA curve represented weight loss of around 9.991 % in the sample at temperature of about 140°C and 16.00 % at 320 °C and 7.772% at 480 °C and 9.375 at 810 °C . This weight loss can be due to the elimination of the water molecules absorbed by the nanoparticles from the atmosphere [24]. Fig. 12 Raman spectrum show the G band is observed at 1594cm^{-1} and the D band is observed at 1347cm^{-1} and Fig. 13 raman spectrum after loading drug show the G band is observed at 1600 cm^{-1} and the D band is observed at 1290 cm^{-1} .We did not observe any change in the bands before and after drug loading, indicating that drug loading did not alter the structural integrity of graphene [25].

Drug Loading and In Vitro Release

The in vitro release profile of GO- MgO-chitosan - cap in PBS solution (pH 7.4) and PH=8 and PH=2.8 (Glycine HCl) is shown in Fig. 14. The total release percentage was around 49.44 % of PH=7.4, 15.52 % of PH=8 and 24.64 % of PH=2.8. The rapid release of cap was probably due to the drug which was adsorbed at or on the surface of the GO [26].

CONCLUSION

In this study, we investigate the potential of GO as an efficient system for loaded drug. Upon successful encapsulation of cap into GO, the anticipated sustained release of cap was achieved. In this study, nanocarriers of graphene oxide and magnesium oxide were fabricated by loading cap and characterizing this carrier using techniques such as FT-IR, XRD, SEM, Zeta potential and TGA. The oral form of cap loaded inside the nanocomposite was evaluated and the release behavior at pH was shown. The study showed that the release amount at pH 7.4 is faster than pH 2.8 and 8. This method is considered promising for oral drug delivery due to the ability of this nanocomposite loaded with cap to control the release.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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