

UNIVERSITI TEKNOLOGI MARA

**SYNTHESIS AND
CHARACTERISATION OF
LAYERED DOUBLE HYDROXIDE-
PALMITIC ACID
NANOCOMPOSITE FOR
CONTROLLED RELEASE AND
ANTIMICROBIAL ACTIVITY
STUDIES**

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ABSTRACT

An effective drug delivery system enhances bioavailability to improve therapeutic outcomes. Incomplete drug administration can reduce effectiveness, requiring higher or more frequent doses. This study focuses on developing nanocomposites that use layered double hydroxides (LDHs) as drug carriers to improve bioavailability. Zinc-aluminium (ZnAl-LDH) and calcium-aluminium (CaAl-LDH) LDHs were synthesized, and palmitic acid (PA) was intercalated into these structures using the co-precipitation method, forming ZnAl-LDH-PA and CaAl-LDH-PA nanocomposites. Powder X-ray diffraction (PXRD) analysis confirmed successful intercalation, with increased interlayer spacing from 8.60 Å to 14.95 Å for 0.05 M CaAl-LDH-PA and from 8.92 Å to 14.35 Å for 0.2 M ZnAl-LDH-PA. Fourier transform infrared (FTIR) spectroscopy further validated PA incorporation by showing the absence of nitrate peaks at 1,347 cm^{-1} and the presence of asymmetrical and symmetrical COO^- stretching vibrations in the 1,540 to 1,577 cm^{-1} range. Energy dispersive X-ray (EDX) analysis confirmed intercalation by detecting no nitrogen, while carbon accounted for 56.8% in CaAl-LDH-PA and 71.9% in ZnAl-LDH-PA. Brunauer-Emmett-Teller (BET) surface area measurements indicated significant increases, from 7.93 m^2g^{-1} to 19.8 m^2g^{-1} for CaAl-LDH-PA and from 4.82 m^2g^{-1} to 21.35 m^2g^{-1} for ZnAl-LDH-PA, confirming anion exchange. Thermal stability assessments demonstrated improved decomposition temperatures, shifting maximum weight loss to 260°C for CaAl-LDH-PA and 220°C for ZnAl-LDH-PA compared from 205°C for pure PA. Controlled release studies of PA confirmed both nanocomposite exhibit pH-dependent release behaviour. CaAl-LDH-PA showing highly sustained at pH 4.8 with a maximum release of 75%, while ZnAl-LDH-PA show the highest release efficiency in pH 4.8 but the most sustained release in pH 7.4. Antibacterial tests showed the intercalation of PA into both nanocomposites preserve the antimicrobial ability of PA against gram-positive and gram-negative bacteria. These findings highlight the potential of LDH-PA nanocomposites as promising carriers for drug delivery applications.

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Nanocomposites have become a revolutionary field of research in materials science as they offer advantages and alternatives to overcome the obstacles of today's engineering materials. A nanocomposite is a multiphase composite material in which one of the phases has a nanoscale morphology or dimensions in the range of 10–100 nm (Sen, 2020). Nanocomposites have many advantages. The most significant advantage is their excellent mechanical properties, as they have improved strength, toughness, and ductility compared to conventional composites due to a high surface-to-volume ratio (Omanović-Miklićanin et al., 2020). In addition, the nanocomposite is considered a versatile and flexible material, as the size, shape, and composition of the nanoparticles can be customised and designed by researchers to exhibit a wide range of properties. This expands the application potential of nanocomposites, which ranges from electronics to medicine.

In the medical field, nanocomposites such as layered double hydroxide (LDH) show great potential due to their advantages, such as biocompatibility, antigenicity, acceleration of tissue regeneration, biodegradability, bioactive interface and controlled release of drugs, especially in drug delivery studies (Izbudak et al., 2021). LDHs are an example of a two-dimensional (2D) nanocomposite and have been described as an anionic clay that exhibits similar properties to naturally occurring brucite ($\text{Mg}(\text{OH})_2$). This compound comprises a positively charged host layer formed by the partial substitution of divalent (M^{2+}) such as Zn^{2+} cations by trivalent (M^{3+}) such as Al^{3+} cations. This positive charge deficit is compensated by anions located in the interlayer space and bound to water molecules (Chaillot et al., 2021). The easily accessible interlayer gives them the ability for anion exchange and ability of organic-inorganic intercalation. These properties have the potential as great carriers for drug delivery systems where the drug can be intercalated and deintercalated into the interlayer of the LDH host.

Palmitic acid (PA), also known as hexadecenoic acid, exhibits a diverse range of applications owing to its notable advantages. Above all, its inherent high stability,