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Mg-Al LAYERED DOUBLE HYDROXIDE NANOPARTICLE-LOADED POLYMERIC MICRONEEDLES FOR ENHANCED DRUG LOADING CAPACITY

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Abstract

Layered double hydroxides (LDHs) have emerged as promising nanocarriers for drug delivery due to their high loading capacity and biocompatibility. In this study, Mg–Al LDH nanoparticles were successfully synthesized and utilized as drug carriers for doxorubicin (DOX) loading. The drug-loaded LDH nanoparticles were subsequently incorporated into polyvinyl alcohol (PVA)-based polymeric microneedles to enhance drug loading performance. The encapsulation efficiency and drug loading capacity of the LDH nanoparticles were determined to be 87.3 ± 0.46% and 43.6 ± 0.23%, respectively, indicating a high affinity between doxorubicin and the LDH structure. The incorporation of LDH nanoparticles into the microneedle matrix resulted in a uniform and stable composite system suitable for transdermal applications. Overall, the developed Mg–Al LDH nanoparticle-loaded PVA microneedles demonstrate significant potential as an efficient platform for high-capacity drug loading. This system provides a promising approach for improving the performance of polymeric microneedle-based drug delivery systems.
Keywords: *LDH nanoparticles, drug delivery, drug loading, encapsulation efficiency, microneedle systems*

Introduction

Efficient drug delivery systems remain a key challenge in modern biomedical research due to limitations of conventional methods, such as low bioavailability, rapid degradation, and limited loading capacity. Layered double hydroxides (LDHs), a class of anionic clay materials with a unique lamellar structure, have emerged as promising nanocarriers owing to their tunable composition, high surface area, and excellent biocompatibility. In particular, Mg–Al LDHs exhibit high structural stability and strong interactions with drug molecules, making them suitable candidates for enhanced drug loading applications (Chubar N., Gerda V., Megantari O.,

Mičušík M., Omastová M., Heister K., Man P., Fraissard J., 2013).

Doxorubicin (DOX) is a widely used chemotherapeutic agent with high efficacy against various cancers; however, its clinical application is limited by systemic toxicity and nonspecific distribution. Therefore, developing delivery systems that enhance its loading capacity and stability is essential Polymeric microneedles have emerged as a minimally invasive and effective drug delivery platform, offering painless administration and ease of use. Polyvinyl alcohol (PVA), a biocompatible and water-soluble polymer, is widely used in microneedle fabrication due to its favorable mechanical properties and safety (Lee J. W., Park J. H., Prausnitz M. R., 2008; Ji J., Tay F. E.H., Miao J., Iliescu C., 2006).

In this study, Mg–Al LDH nanoparticles were synthesized and employed as carriers for DOX loading. The drug-loaded LDH nanoparticles were subsequently integrated into PVA-based polymeric microneedles to enhance drug loading capacity. The encapsulation efficiency and drug loading performance of the developed system were systematically evaluated, demonstrating its potential as an effective platform for advanced drug delivery applications.

Materials and methods

Preparation of Doxorubicin Standard Solutions and Calibration Curve. The encapsulation efficiency and drug loading capacity of LDH nanoparticles were evaluated using doxorubicin (DOX) as a model drug. A stock solution of DOX (0.5 mg/mL) was prepared in deionized water, followed by serial dilution to obtain standard solutions (1–100 μg/mL). The absorbance of each solution was measured at 480 nm using a UV–Vis spectrophotometer. All measurements were performed in triplicate, and a calibration curve was constructed by plotting absorbance versus concentration to obtain a linear regression equation for quantitative analysis.

Drug Loading into Mg-Al LDH Nanoparticles. DOX loading into Mg-Al LDH nanoparticles was performed via an adsorption–intercalation mechanism. Briefly, 50 mg of LDH was dispersed in 50 mL of DOX solution (0.5 mg/mL), followed by ultrasonication for 10 minutes to ensure uniform dispersion. The suspension was then stirred at room temperature for 24 h under dark conditions. The pH was adjusted to 4.5–5 using 0.1 M HCl. After incubation, the mixture was centrifuged at 10,000 rpm for 10 min, and the precipitate was washed three times with deionized water to remove unbound drug.

Determination of Encapsulation Efficiency and Drug Loading Capacity. The amount of DOX loaded into LDH nanoparticles was determined using an indirect method. The concentration of free DOX in the supernatant and washing solutions was measured by UV–Vis spectroscopy at 480 nm. The total free drug was calculated as the sum of DOX in all fractions, and the loaded amount was obtained by subtracting it from the initial DOX amount. Encapsulation efficiency (EE%) and drug loading capacity (DL%) were calculated using standard equations.

Result and discussions

In recent years, nanoscale carriers such as nanomicelles, liposomes, metal–organic frameworks (MOFs), and LDHs have been extensively developed to improve drug delivery systems. These materials offer high surface area, tunable structures, and efficient drug encapsulation. MOFs, in particular, have attracted attention due to their porous three-dimensional structures and high loading capacity, enabling efficient drug encapsulation and controlled, stimuli-responsive release. However, their limited stability in aqueous and physiological conditions, as well as potential cytotoxicity, restrict their practical application. In contrast, LDH-based nanocarriers exhibit superior biocompatibility, structural stability, and ease of synthesis. Their lamellar structure enables effective drug intercalation, making them promising alternatives for drug delivery applications (Horcajada P., Chalati T., Serre C., Gillet B., Sebrie C., Baati T., Eubank J. F., Heurtaux D., Clayette P., Kreuz C., Chang J., Hwang Y. K., Marsaud V., Bories P., Cynober L., Gil S., Férey G., Couvreur P., Gref R., 2009).

LDH nanoparticles possess a lamellar structure composed of positively charged hydroxide layers and interlayer anions, enabling effective drug intercalation. This feature makes them highly suitable for drug delivery applications. Previous studies have

shown that LDH systems can efficiently load anticancer drugs, protect them from degradation, and provide controlled release, mainly through an ion-exchange mechanism. In addition, LDHs exhibit excellent biocompatibility, low toxicity, and high stability in biological environments. These properties make LDH nanoparticles promising candidates for enhancing drug loading capacity and improving the performance of drug delivery systems (Yu J., Wang Q., O'Hare D., Sun L., 2017).

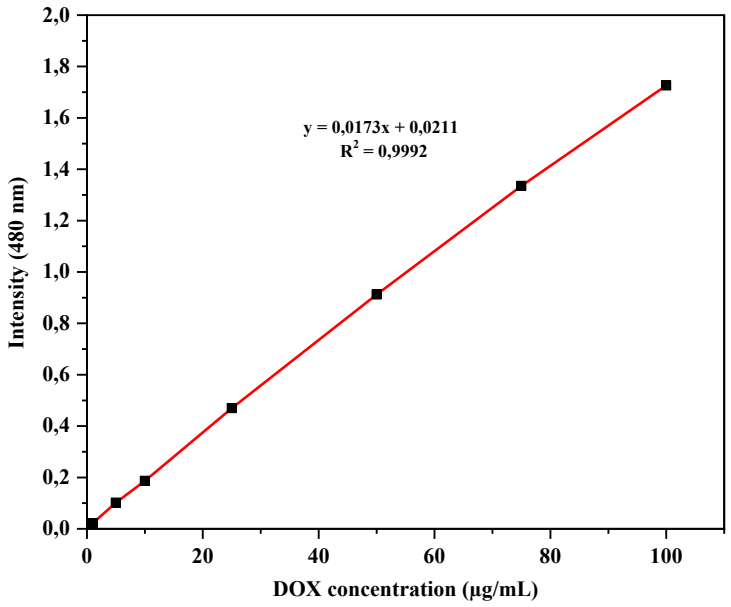
DOX loading into LDH nanoparticles was performed via an adsorption–intercalation mechanism using different drug-to-carrier ratios (1:1, 2:1, and 3:1). The results showed that this ratio significantly influences nanoparticle formation and loading performance. At a 1:1 ratio, heterogeneous particle sizes were observed, indicating insufficient interaction between DOX and LDH layers. In contrast, a 3:1 ratio led to particle aggregation due to excess drug disrupting structural integrity. The optimal results were obtained at a 2:1 ratio, which provided uniform morphology, good dispersion, and enhanced stability. After 24 h of stirring, a homogeneous reddish suspension was observed, confirming successful intercalation of DOX into LDH layers. These findings demonstrate that the drug-to-carrier ratio is a key factor affecting both loading efficiency and nanoparticle stability.

Following lyophilization, LDH–DOX nanoparticles were obtained as a dry powder, which readily redispersed in water to form a stable colloidal system without aggregation. This indicates excellent physicochemical stability and hydrophilicity, which are essential for biomedical applications. The obtained nanoparticles were further characterized in terms of hydrodynamic size, polydispersity index (PDI), and morphology to evaluate dispersion quality and stability. These parameters are crucial as they influence cellular uptake and therapeutic efficiency. In addition, encapsulation efficiency and drug loading capacity were analyzed as key indicators of drug loading performance, confirming the effectiveness of the LDH–DOX system.

The concentration of DOX in the supernatant and washing solutions after centrifugation was determined using UV–Vis spectroscopy at a wavelength of 480 nm. A calibration curve constructed from standard DOX solutions (see Fig. 1) exhibited excellent linearity, with a correlation coefficient of $R^2 = 0.9992$, confirming the reliability of the analytical method.

The amount of free (unloaded) DOX was determined by measuring the absorbance of the supernatant and all washing solutions. The total free drug content was calculated as the sum of DOX present in these fractions and was found to be 3.195 mg.

Figure 1. *Calibration curve for DOX constructed from UV spectral values obtained at an emission wavelength (λem) of 480 nm*



Conclusion

In this study, Mg–Al LDH nanoparticles were successfully used as nanocarriers for DOX loading, achieving high encapsulation efficiency (87.3 ± 0.46%) and drug loading capacity (43.6 ± 0.23%). These results highlight the strong potential of LDH nanoparticles as effective carriers for cytostatic drugs. The high loading performance is attributed to the layered structure of LDH and strong electrostatic interactions with DOX molecules, enhanced under optimized pH conditions. The presence of small amounts of DOX in the supernatant and washing solutions indicates both surface adsorption and intercalation mechanisms. The low standard deviation values confirm the high reproducibility of the method, demonstrating that LDH-based nanocarriers provide a stable and efficient platform for high-capacity drug loading.

References

Chubar N., Gerda V., Megantari O., Mičušík M., Omastová M., Heister K., Man P., Fraissard J. (2013). Applications versus properties of Mg–Al layered double hydroxides provided by their syntheses methods: Alkoxide and alkoxide-free sol–gel syntheses and hydrothermal precipitation, Chem. Eng. J. – 234. – P. 284–299. URL: URL: https://doi.org/10.1016/J.CEJ.2013.08.097.

Mallakpour S., Azadi E., Hussain C. M. (2021). Recent advancements in synthesis and drug delivery utilization of polysaccharides-based nanocomposites: The important role of nanoparticles and layered double hydroxides, Int. J. Biol. Macromol. – 193. – P. 183–204. URL: https://doi.org/10.1016/j.ijbiomac.2021.10.123.

Lee J. W., Park J. H., Prausnitz M. R. (2008). Dissolving microneedles for transdermal drug delivery, Biomaterials – 29. – P. 2113–2124. URL: https://doi.org/10.1016/j.biomaterials.2007.12.048.

Ji J., Tay F. E.H., Miao J., Iliescu C. (2006). Microfabricated microneedle with porous tip for drug delivery, J. Micromechanics Microengineering – 16. – P. 958–964. URL: https://doi.org/10.1088/0960–1317/16/5/012.

Horcajada P., Chalati T., Serre C., Gillet B., Sebrie C., Baati T., Eubank J. F., Heurtaux D., Clayette P., Kreuz C., Chang J., Hwang Y. K., Marsaud V., Bories P., Cynober L., Gil S., Férey G., Couvreur P., Gref R. (2009). Delivery and imaging, Nat. Mater. – 9. – P. 172–178. URL: https://doi.org/10.1038/nmat2608.

Yu J., Wang Q., O'Hare D., Sun L. (2017). Preparation of two dimensional layered double hydroxide nanosheets and their applications, Chem. Soc. Rev. – 46. – P. 5950–5974. URL: https://doi.org/10.1039/c7cs00318h.

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