

# **Stearate intercalated layered double hydroxides: methods and applications**

by

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## Declaration

I, Edith Phyllis Landman, declare that this thesis is my own work, except where specifically acknowledged in the text. I also declare that neither this thesis nor any part thereof has been submitted by me for degree purposes at any other university.

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E.P. Landman

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## Abstract

Stearate anions were successfully intercalated into the layered double hydroxide  $Mg_4Al_2(OH)_{12}CO_3 \cdot 3H_2O$  (LDH- $CO_3$ ) by several methods to form LDH-SA. The intercalation method which involved the acid-base reaction between emulsified stearic acid (SA) and the carbonate anions in aqueous media was studied for the first time. This method led to the formation of more LDH-SA than well known methods such as melting the carboxylic acid in the presence of the LDH, allowing the interlayer region to swell in the presence of glycerol and reconstructing the calcined LDH in the presence of aqueous sodium stearate. Other literature methods involve ion-exchange of  $Cl^-$  in LDH-Cl with stearate in aqueous sodium stearate, usually under  $N_2$  atmosphere. The methods developed in this study are more industrially viable because the more easily produced LDH- $CO_3$  is used and no  $N_2$  atmosphere is necessary.

The LDH-SA was successfully used to intercalate sodium polyvinyl sulphonate by an ion exchange with the intercalated stearate, without the need for a  $N_2$  atmosphere. This method of production could be useful for the production of nanocomposites in general, for example anionic polymer chains (such as DNA) and anionic clays.

The same intercalation reaction was allowed to take place *in situ* during the formation of dextrin-alginate-glycerol film solutions in water-ethanol media. The stearate intercalated as a bilayer in the interlayer region of the LDH. The SA to LDH ratio was varied from 100% SA to 100% LDH. Around the middle of the series a minimum water vapour permeability (WVP) was obtained, which corresponded to an 80% reduction in WVP in comparison to the reference (blank) film. Around the middle of the series a maximum increase in Young's modulus, corresponding to a 213% increase in comparison to the blank film, was obtained. Around the middle of the series a reduction in the intensity of the basal reflection and interlayer distance showed that some exfoliation (delamination) took place.

## Samevatting

Verskeie metodes om stearaatione tussen die hidroksiedlae van  $Mg_4Al_2(OH)_{12}CO_3 \cdot 3H_2O$  (LDH- $CO_3$ ) te laat inskuif, was getoets. Die metode wat gebaseer is op die suur-basis inskuifreaksie tussen geemulgeerde steariensuur (SA) en die karbonaatanione in watermedium is vir die eerste keer getoets. Hierdie metode het gelei tot die vorming van meer LDH-SA as welbekende metodes soos smelting van die karboksielsuur in die teenwoordigheid van die LDH, die swelling van die interlaagruimte deur middel van gliserol asook die herkonstruksie van die gekalsineerde LDH in die teenwoordigheid van waterige natriumstearaat. Ander literatuurmetodes behels die ionuitruiling van die  $Cl^-$  van LDH-Cl met stearaat in waterige natriumstearaat, gewoonlik onder 'n stikstofatmosfeer. Die metodes wat tydens hierdie studie ontwikkel is, is meer industrieel toepasbaar omdat die maklik produseerbare LDH- $CO_3$  benut word en ook geen stikstofatmosfeer vereis word nie.

Die LDH-SA is suksesvol aangewend om die inskuif van natriumpolivinielsulfonaat te bewerkstellig deur 'n ionuitruilmeganisme met die geinterkaleerde stearaatione, sonder dat 'n stikstofatmosfeer benodig is. Hierdie metode kan in die algemeen aangewend word om nano-saamgestelde materiale te vervaardig vanuit enige anioniese polimeer (bv. DNS) en anioniese kleie.

Dieselfde inskuifreaksie is toegelaat om plaas te vind tydens die vorming van 'n dekstrien-alginaat-gliserol gebaseerde filmvormende oplossing in 'n water-etanol medium. 'n Dubbellaag stearaat het in die LDH tussenlaagruimte ingeskuif. Die hoeveelheid SA tot LDH was gevarieer van 100% SA tot 100% LDH. 'n 80% verlaging in waterdamp permeabiliteit was verkry in die middel van die reeks in vergelyking met die verwysingsfilm (kontrole). In daardie omgewing is ook 'n maksimum toename in Young's modulus van ongeveer 213% van die kontrole film waargeneem asook 'n daling in die intensiteit van die basale refleksie en interlaagafstand, wat aandui dat die LDH-SA in 'n mate ontblaar het.



### **1 Corinthians 2:2**

For I determined not to know any thing among you, save Jesus Christ, and him crucified.

### **1 Corinthians 1:17-21**

For Christ sent me not to baptize, but to preach the gospel: not with wisdom of words, lest the cross of Christ should be made of none effect. For the preaching of the cross is to them that perish foolishness; but unto us which are saved it is the power of God. For it is written, I will destroy the wisdom of the wise, and will bring to nothing the understanding of the prudent. Where *is* the wise? where *is* the scribe? where *is* the disputer of this world? hath not God made foolish the wisdom of this world? For after that in the wisdom of God the world by wisdom knew not God, it pleased God by the foolishness of preaching to save them that believe.



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## Nomenclature and Abbreviations as used by the Author

Term	Abbreviation	Explanation
Hydrotalcite	HT	The layered double hydroxide of composition $Mg_6Al_2(OH)_{16}CO_3 \cdot 4H_2O$ .
Layered double hydroxide	LDH	Anionic clay minerals consisting of $Mg(OH)_2$ like layers which contains both divalent and trivalent cations in different ratios.
Interlayer		The region between the two hydroxide layers in which the charge balancing anions and crystal water lie.
Sand-rose morphology		Particles consisting of many intergrown smaller particulates which reduce the surface area.
Intercalation		Process in which atoms, ions or molecules are trapped between layers in a crystal lattice without the formation of a formal chemical bond [1].
Intercalate (verb)		Diffusion of atoms, ions or molecules into the interlayer region of a layered compound.
Intercalate (noun) or Intercalation compound		A compound in which atoms, ions or molecules are trapped between layers in a crystal lattice [1].
Adsorption		Formation of (a) layer(s) of gaseous, liquid or solid molecules on the surface of a molecule, held by chemical or physical forces [1].
Stearic acid	SA	A solid saturated fatty acid, $CH_3(CH_2)_{16}COOH$ [1].
Carboxylic acid		Organic compounds containing the group $-COOH$ [1].





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Fatty acid	FA	An organic compound consisting of a hydrocarbon chain and a terminal carboxyl group [1].
Ion exchange		The exchange of ions of the same charge between a solution and a solid in contact with it [1].
Anionic exchange capacity	AEC	The amount of exchangeable anions available within the crystal structure of an adsorbent material, expressed in mmol/g or mmol/100 g adsorbent.
Brønsted acid/base		A Brønsted-Lowry acid is a substance that donates a proton and a Brønsted-Lowry base is a substance that accepts a proton [2].
Lewis acid/base		A Lewis acid is a substance that accepts an electron pair, and a Lewis base is a substance that donates an electron pair [2].
Carbonate layered double hydroxide	LDH-CO <sub>3</sub>	A layered double hydroxide into which the carbonate anion has intercalated.
Chloride layered double hydroxide	LDH-Cl	A layered double hydroxide into which the chloride anion has intercalated.
Stearate layered double hydroxide	LDH-SA	A layered double hydroxide into which the stearate anion has intercalated.
Emulsion		A kinetically stable dispersion of two immiscible liquids, stabilized by the presence of a surfactant at the interface.
Emulsifier		A surfactant that resides at the interface of two immiscible liquids in order to stabilize the mixture of the two liquids.

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Micro-emulsion		A thermodynamically stable mixture of two immiscible liquids, stabilized by a high concentration of surfactant and a co-surfactant.
Surfactant		Molecules with both hydrophilic and hydrophobic parts.
Sodium dodecyl sulfate	SDS	An anionic surfactant, $\text{CH}_3(\text{CH}_2)_{11}\text{SO}_4\text{Na}$
Calcine		Heat a substance to an elevated temperature to facilitate a reaction, for example decomposition reaction. In the case of the LDHs used in this study, the calcination facilitates the dehydration, decarbonation and dehydroxilation reactions which can collectively be described as decomposition reactions.
Reconstruction		Rehydration of a calcined LDH in the presence of an aqueous solution of an anion.
Layered double oxide	LDO	Calcined LDH.
Poly(sodium vinylsulfonate)	PVS	The sodium salt of a polymer consisting of vinyl sulfonic acid monomeric units.
Polyanion or anionic polymer		A polymer consisting of anionic (negatively charged) monomeric units.
Polyelectrolyte		A polymer consisting of positively or negatively charged monomers
Cross linkage		A short side chain of atoms (or a charged moiety) linking two longer chains in a polymeric material [1].
Sodium alginate		The sodium salt of alginic acid, a polysaccharide occurring in the cell walls of brown algae [1].

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Dextrin	An intermediate polysaccharide resulting from the hydrolysis of starch by amylase enzymes or acids [1].
Polysaccharide	Polymers consisting of long chains of monosaccharide (simple-sugar) molecules [1].
Plasticizer	Materials incorporated into plastic resins to change workability, flexibility, flow and impact resistance [3], for example glycerol.
Glycerol	1,2,3-trihydroxypropane, used as a plasticizer in thermoplastic starch [3].
Defoamer	A substance (usually petroleum oil based) that prevents a surfactant from creating foam in a mixture.
Filler	Solid inert material added to a polymeric matrix to change its physical properties or to dilute it for economy [1].
Composite material	A material consisting of two or more substances.
Nanocomposite	A composite material in which one of the constituents have at least one dimension which is lower than 100 nm.
Foliate	With regard to polymer chains or layered compounds, whereby polymer chains line up or the individual layers are stacked together.
Exfoliate	Polymer chains or individual inorganic layers that are dispersed randomly within a composite.

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Exfoliation		Process whereby polymer chains that were aligned or stacked inorganic layers are pushed apart into a random pattern.
Viscosity		A measure of the resistance to flow that a fluid offers when it is subjected to shear stress [1].
Water vapour permeability	WVP	The mass of water vapour that moves through a film of certain thickness per unit time, unit area and unit difference in water vapour pressure.
Young's modulus		Synonym for the modulus of elasticity, namely the ratio of the unit stress to the unit strain [4].
Tensile strength		Synonym for ultimate strength, namely the maximum ordinate to the stress-strain curve [4].
Percentage strain (elongation) at break		The increase in length of the gage after fracture divided by the initial gage length and multiplied by 100 [4].
Retrogradation		A process which happens during the ageing of starch in which the starch chains lose water and form intermolecular hydrogen bonds.
Crystalline		Used to indicate that a chemical species has long-range structure.
Amorphous		Used to indicate that a chemical species does not have long-range structure. This does, however, not preclude the existence of short-range structure.
X-ray diffraction	XRD	Diffraction of X-rays by a crystal due to the comparable size of the wavelengths of X-rays and the distances between atoms in crystals [1].

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X-ray fluorescence	XRF	The emission of X-rays from excited atoms produced by the impact of high-energy electrons, particles or a primary beam of X-rays. The wavelengths of the fluorescent X-rays are measured by an X-ray spectrometer as a means of chemical analysis [1].
Scanning electron microscopy	SEM	A beam of primary electrons scans the specimen surface and those that are reflected, together with secondary electrons emitted, are collected to form a visual image [1].
Transmission electron microscopy	TEM	A sharply focused electron beam passes through a thin section of metal-coated specimen onto a fluorescent screen to form a visual image [1].
Freeze drying		A process used to dehydrate heat sensitive substances whereby the product is deep-frozen and the ice is sublimed by reducing the pressure [1].
Freeze fracturing		A process whereby a material is embrittled by freezing it in liquid nitrogen and broken to obtain a clean surface without smearing.
Microscopic structure		The shape of a particle as observed with a microscope, using either an optical or an electron microscope.
Fourier Transform infrared spectroscopy	FTIR	Fourier transformations are done on infrared spectroscopic data by a computer to enable the intensity against wave number to be plotted with high sensitivity [1].

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Thermogravimetry	TG	The mass of a sample in a controlled atmosphere is recorded continuously as a function of temperature or time [5].
Derivative of the thermogravimetric trace	DTG	Derivative of the thermogravimetric curve

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## **Preface**

The research done for this PhD study is joined in this book in the form of 3 extended self-contained articles (Chapters 2-4) featuring different aspects of the research. Chapter 1 is an introduction to the general concepts dealt with in this research as well as an outline of the aims and importance of this research. Chapter 5 is a general concluding chapter, with some ideas on future research in this field. The abbreviated versions of the articles were submitted to different journals. A list of publications, patents and conference proceedings emanating from this research are given in Appendix A.