The present research is aimed at synthesis, dispersion, coating and toxic gas reactivity of Layered Double Hydroxides (LDHs) and their different composites. In order to synthesize and develop composite materials containing LDHs, different approaches were taken up viz., by clay-LDH aqueous gel, SiO₂-LDH non-aqueous ‘sol-gel’ and organic-inorganic polymer-LDH hybrid gel route. The focal theme of the work undertaken in the present Ph.D. thesis was the synthesis of different dispersible composites by LDHs, effect of conditions of synthesis and compositional factors on the stability and flow behaviour of the composite dispersions, the effect of compositional variations on coating and film formation properties and efficacy of such coated substrates in the abatement of environmental pollution by different toxic gases like N₂O, H₂S, SO₂.

The characterization of the neat LDH and LDH based composites were carried out by powder X-ray diffraction study, FT-IR, thermal analysis (DTA-TGA-DTG), flow behaviour study of the dispersions containing LDH and its derived composite gels were carried out by the controlled stress rheometer. The electrokinetic mobility or zeta potential measurement of the flowable dispersions were carried out with Laser Doppler Velocimetry technique at 25°C under a 10 mW He–Ne laser (M/S Malvern Instruments Zetasizer 3000-HS).
Abstract

The coating of the suitable dispersions over different solid substrates was carried out in a dip-coater with programmed dipping and withdrawal sequence to get thin crack free films. Finally, the efficacy of the powdered as well as coated materials were studied in certain ‘Gas-Solid’ reactions where the LDHs and their derived composites act either as catalyst or adsorbent for treatment of environmentally harmful gases like N₂O, H₂S, SO₂.

The content of this thesis is distributed over six chapters followed by conclusion, references. Each chapter is virtually complete in itself including figures and references.

The Chapter-1 deals with an introductory overview focusing the background and explaining the motive of undertaking the present work.

The Chapter-2 deals with review of literature, the current status as well as the historical developments in the field of LDH, its structure, properties and its various applications have been highlighted in this chapter.

The Chapter-3 represents a description of experimental methods, chemicals and materials and details of equipments used for physical measurements.

The Chapter-4 describes the synthesis of LDHs viz., Mg-Al, Ca-Al, Ni-Al, Zn-Al, Zn-Cr and Cu-Cr in alkaline and acidic pH. It also deals with the characterization of the prepared LDHs by spectral and crystallographic techniques. The chemical formulae of the prepared LDHs were derived by different techniques (like AAS, ICP-AES, Ion Chromatography and CHN analysis). The chemical formulae of the prepared
LDHs were assigned following the method by Prof. Rudolf Allmann, University of Marburg.

The thermal behaviour of the pristine LDHs and the Clay-LDH aqueous composites were studied by TGA-DTG-DTA techniques. The flow behaviour study of the composite gels of LDH with clay in aqueous medium was carried out at different reaction conditions in the rheometer.

The morphological features of the composites were obtained from SEM studies. The mechanical strength of the coated α-alumina substrates with composite gels at different ratios of clay and LDH along with their pristine forms (clay and LDH) were studied in Universal Testing Machine.

The ‘Gas-Solid’ reaction studies for treatment of toxic N₂O, H₂S and SO₂ were carried out with LDH and its clay composites in aqueous medium. For N₂O decomposition study Ni-Al and Mg-Al LDHs and their clay based composites were used. For SO₂ adsorption study Ca-Al and Mg-Al LDH and their clay derived composites were used. For H₂S adsorption study Zn-Al LDH and clay derived composites were used.

The Chapter-5 deals with synthesis of LDH (Ni-Al and Mg-Al) prepared by soft chemical or so called ‘sol-gel’ approach using acetylacetonate and alkoxide precursors.

The composites of Ni-Al and Mg-Al LDHs were synthesised supporting over silica at different ratios of the constituents. The characterization of LDHs and their silica
derived composites in non-aqueous medium were carried out by X-ray study, spectral techniques (FT-IR), thermal analysis (TGA-DTG-DTA). The morphological characterization was carried out by SEM.

Finally ‘Gas-Solid’ reactions were carried out using Mg-Al LDH and its silica derived composites for decomposition of N₂O.

The Chapter-6 deals with synthesis of hybrid inorganic-organic gels of Ni-Al and Zn-Cr LDHs with acrylamide monomer. The flow behaviour study of the hybrid gels exhibited flowable nature of the prepared gels suitable for coating over solid preforms.

The intercalated acrylamide monomers showed in-situ intercalative polymerization in the interlayer slabs of the inorganic host material (LDHs). The polymerization of the acrylamide monomers inside the inorganic host layers of LDHs was further corroborated by the FT-IR studies where the peak near 1110 cm⁻¹ in both the hybrid composite gels correspond to \( \nu_4 (C-C) \) asymmetric stretching mode of polyacrylamide. The morphological study in hybrid composite gels by SEM showed that agglomerated nature of the pristine LDHs particles disappeared in the hybrid composite gel. The hexagonal shaped LDH particles were clearly visible in the TEM analysis of the dried hybrid gels where the LDH particles were well exfoliated in the polymer matrix of the acrylamide.

The conclusion provided at the end highlights the salient findings of the research work. Part of the work described in the thesis has been published or under communication (as detailed later) with the rest being processed for publication.
Abstract

Published/Accepted


Seminars/Conferences


Abstract


Communicated

