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Research paper

Facile preparation approach of nanocomposite based on water-soluble polymer and layered double hydroxides for the enhancement of leather dyeing

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ABSTRACT

LDH/P(DMDAAC-AA-SAS) nanocomposites with LDH loading of 0.5, 1.0, 1.5, 2.0, 2.5, and 3.0% have been prepared by facile approach using water-soluble polymer dimethyl diallyl ammonium chloride-acrylic acid-sodium allyl sulfonate [P(DMDAAC-AA-SAS)] and layered double hydroxides (LDH) through ultrasound and self-assembly process. X-ray diffraction (XRD) and transmission electron microscope (TEM) of the nanocomposites show that the LDH are partly exfoliated, and the solubility of the LDH/P(DMDAAC-AA-SAS) can be affected by the LDH loading amount. Fourier transform infrared spectrometer (FTIR) analysis indicates that the nanocomposites are prepared. Different LDH loading amount LDH/P(DMDAAC-AA-SAS) nanocomposites have been applied in leather process. The results show that, the 'L' values of the 1 wt% LDH loading LDH/P(DMDAAC-AA-SAS) nanocomposite treated black leather can be reduced to 21.56 while the control leather is 25.22, which indicating the better dyeing properties of LDH/P(DMDAAC-AA-SAS) nanocomposite. In addition, 1 wt% LDH loading LDH/P(DMDAAC-AA-SAS) nanocomposite resulted in increment of leather thickening rate by 27.4% than control leather and physical characteristics of leather treated by nanocomposite can also be improved.

1. Introduction

Leather production is a process that converted hide/skin into useful materials to meet the different demands of human being (Orlita, 2004). Usually, the key process for leather production is tanning, which renders permanent stability to the leather products.

Nowadays between 80% and 90% of leathers are tanned with trivalent chromium salts due to their advantages over other tanning agents (Chattopadhyay et al., 2012). But the chrome tanning wastewater is associated with serious environmental impacts including contamination of soil (Tariq et al., 2009). However, not only chromium in the tanning waste, there are also large amounts of chloride ions. Before tanning, the pH of hide/skin is lowered to about 2.5–3.0% by using acid, and the process is known as "pickling". While pickling, 6–8% common salt is added to avoid the swelling of the hide/skin, so the waste water also contain a lot of chloride ions. Polymer with strong anionic groups has good effects to avoid the swelling of hide (Suresh et al., 2001), but the anionic groups may result in a negative effect to the penetration and combination of anionic dyes with leather, as it known as "colour lose" effect. To solve these problems, amphoteric polymer PDM-AA-SAS was prepared in our previous study and it could achieve low-salt pickling and less chrome tanning process (Jia et al., 2016), but the dyeing properties cannot be improved.

Layered double hydroxides (LDH) are a class of ionic lamellar compounds made up of positively charged brucite-like with an interlayer region containing charge compensating anions and solvation molecules (Wang and O'Hare, 2012). The chemical composition of a LDH is often described by the general formula $[M^{II}_{1-x}M^{III}_{x}(OH)_2]^{x+}[A^{n-}_{x/n}yH_2O]^{x-}$, where M^{II} and M^{III} are divalent and trivalent metal cations and A^{n-} is an-valent anion (Kovanda et al., 2010; Özgümüş et al., 2013). Nowadays, LDH can be widely used in different filed, such as catalyst (Xu et al., 2011), ion exchange hosts (Millange et al., 2000), drug delivery hosts (Alcantara et al., 2010), fire retardant additives (Manzi-Nshuti et al., 2009), polymer thermal stabilizer (Liu et al., 2008), UV light shielding (Cao et al., 2013), enhancement of mechanical property (Hu and Chen, 2014a,b), photoluminescence (Wu et al., 2017), rheology (Hu and Chen, 2014a,b) and so on. LDH are also well known ion-exchangers/adsorbents for dyes (Chakraborty et al., 2010). Sujata Mandal et al. synthesized an aqueous dispersion of layered double hydroxide, and explored it as dye leveling agent for enhanced

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colour intensity and consistency in leather dyeing process (Mandal et al., 2015). Since the leather making is carried out in water system, the dispersion of LDH in aqueous restricts its application in leather process (Abellan et al., 2014). How to get a stable water-based LDH dispersed system is important.

LDH based nanocomposite can be prepared by various strategies, such as blending (Wang et al., 2013), in-situ (Guo et al., 2013), exfoliation-adsorption (Li et al., 2003) et al. Most of the methods need special conditions such as melting or dissolve the polymer, nitrogen protection or preparation exfoliation LDH nanosheets for the first. This article provide a facile preparation approach of nanocomposite based on water-soluble polymer and LDH by ultrasonic blending, which providing suitable conditions for the assembly of the polymer and the LDH.

Highly dispersed LDH platelets are desirable to maximize the interfacial area with the surrounding polymer. Compatibility of the LDH and polymer can be increased by electrostatic interactions (which can be seen as self-assembly process) or chemically grafting molecules onto the surface of the particles (Kovanda et al., 2010). Hongliang Kang (Kang et al., 2014) tried to disperse and stable LDH nanosheets in an alginate aqueous solution; Cyril Veschambres (Veschambres et al., 2016) reported on the use of waterborne latex and LDH to prepare LDH polymer nanocomposite films by self-assembly process through efficient electrostatic interactions, leading to the formation of a homogeneous cellular network of LDH layers within the material. The advantage of the water solution is the miscibility with hydrophilic LDH, which may facilitate the formation of intercalated or exfoliated LDH/polymer nanocomposites.

In leather making process, different nanomaterials have been introduced into polymer, such as clay (Bao et al., 2009), ZnO (Ma et al., 2014), SiO_2 (Fan et al., 2004), graphene oxide (Lv et al., 2015), et al. Leather collagen can be modified by these nanomaterial in varying degrees, and the properties of leather product can be improved. However, nanocomposites of LDH loading used in leather making process are rarely reported.

In our previous study, the water-based polymer dimethyl diallyl ammonium chloride-acrylic acid-sodium allyl sulfonate shortened as P (DMDAAC-AA-SAS) was prepared via free radical polymerization. The polymer P(DMDAAC-AA-SAS) was applied in leather process, and the process achieved better environmental benefits (Jia et al., 2016). To improve the dyeing properties of leather products, we describe the synthesis of water-soluble nanocomposite by positively charged LDH layers and negatively charged P(DMDAAC-AA-SAS) through facile ultrasonic and self-assembly process. The morphology, thermal properties of the LDH/P(DMDAAC-AA-SAS) nanocomposites are discussed in this study. The application properties of the LDH/P(DMDAAC-AA-SAS) in leather process are also investigated.

2. Materials and methods

2.1. Material

Layered double hydroxides (LDH, Mg_2Al-CO_3^{2-}-LDH, pI 12.6, Beijing University of Chemical Technology). Dimethyl diallyl ammonium chloride (DMDAAC, 60%, Shandong Luyue Chemical Industry Co. Ltd., China), acrylic acid (AA, AR, Tianjin Fuchen Chemical Reagents Factory), sodium allyl sulfonate (SAS, AR, Chengdu Huaxia Reagent Co., LTD), ammonium persulfate (APS, AR, Tianjin Hengxing Chemical Reagent Manufacturing Co. Ltd., China), sodium hydrogen sulfite ($NaHSO_3$, AR, Tianjin Tianli Chemical Reagent Co. Ltd., China), all the reagents were used as received. Basic chrome sulfate (BCS) (33% basicity) Tankrom-AB was supplied by Turkey KROMSAN group was used as chrome tanning agent.

2.2. Synthesis of P(DMDAAC-AA-SAS)

Synthesis of P(DMDAAC-AA-SAS) was prepared as described previously (Jia et al., 2016). Under continuous stirring, monomer mixture DMDAAC (60%, 56.34 g) and AA (36 g) were poured into a 250 mL three-neck-flask equipped with a digital agitator and a reflux condenser in a water bath at 80 °C. Initiator solution (I) was obtained by dissolving 6.79 g APS in 67.9 g deionized water; initiator solution (II) was added 1.70 g $NaHSO_3$ in 17 g deionized water. SAS solution was obtained by dissolving 15.06 g SAS in 8 g deionized water. Initiator solution (I), (II) and 102.4 g deionized water were added in the flask, and SAS solution was added dropwise into the reaction mixture for 0.5 h. The pH of the P(DMDAAC-AA-SAS) solution was 0.6 after 4 h polymerization, then the pH of the P(DMDAAC-AA-SAS) solution was adjusted to 5.0 and 12, respectively. GPC: Mn [348 (PDI: 2.418); 2.8×10^4 (PDI: 2.090)]. Solid content, 30%.

2.3. Preparation of LDH/P(DMDAAC-AA-SAS) nanocomposites

Under continuous stirring with 400 rpm, P(DMDAAC-AA-SAS) solution of different pH (40 g) was poured into a 100 mL three-necked flask equipped with a digital agitator in a water bath at 40 °C, 1% LDH (based on polymer solution weight) was added in the flask. After stirring for 4 h, the mixture was dispersed by 360 W power ultrasonic for 50 min, LDH/P(DMDAAC-AA-SAS) nanocomposites of different pH value were obtained. LDH/P(DMDAAC-AA-SAS) nanocomposites with different LDH loading amount viz., 0.5%, 1.0%, 1.5%, 2.0%, 2.5%, 3.0% (based on polymer solution weight) were also prepared.

2.4. Application in leather process

LDH/P(DMDAAC-AA-SAS) with different LDH loading amounts and P(DMDAAC-AA-SAS) were applied in leather process for experimental trials. The bated pelts were treated with P(DMDAAC-AA-SAS) and LDH/P(DMDAAC-AA-SAS) for 2 h, then attain pH to 3.2 slowly by formic acid, then added 4% BCS drumming for 2.5 h and 0.5% sodium formate drumming for 0.5 h, finally the basified the bath pH to 4.1. The bated pelt pickling and tanning use 4% BCS as control. The post tanning processes for the tanned leathers were the same according to the cattle shoe upper leather production process.

2.5. Characterization

DV-II + viscometer (BROOKFIELD America) was used to test the rotary viscosity of the composite. The P(DMDAAC-AA-SAS) solution and LDH/P(DMDAAC-AA-SAS) nanocomposites were washed with ethanol three times, and then dried at 102 °C for 6 h. Fourier transform infrared spectrometer (FTIR) analysis was performed by FTIR-5DX FTIR (Nicolet, America). X-ray diffraction (XRD) was used to determine the crystal structure of samples. The X-ray diffraction pattern was recorded in the range of 2θ between 5°and 70°. XRD measurement was carried out on D/max-2200 X-ray diffract remoter (Rigaku, Japan) using Cu Ka radiation. The morphologies of LDH/P(DMDAAC-AA-SAS) nanocomposites were studied by a transmission electron microscope (TEM) Tecnai G2 F20 S-TWIN (FEI, America). Thermogravimetric analyses (TGA) were performed using a TGA Q500 (TA, America) analyzer. Samples were heated from room temperature to 600 °C at a heating rate of 20 °C/min under N_2 atmosphere.

2.6. Wet blue analysis

The thickening rate of wet blue was tested by leather thickness tester (MH-YDI Sunshine Electronic Research Institute of Shaanxi University of Science and Technology).

2.7. Crust leather properties analysis

All the crust leather samples were conditioned at standard atmospheric over a period of 48 h before test. For strength properties test,

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