Regular Article

ORGANIC CHEMISTRY RESEARCH
Published by the
Iranian Chemical Society
www.orgchemres.org

Org. Chem. Res., Vol. 3, No. 1, 95-102, March 2017.

Layered Double Hydroxides (LDHs): An Efficient Catalyst System for the Synthesis of Chiral Aminonitriles

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(Received 4 March 2017, Accepted 1 August 2017)

Strecker reaction is one of the simplest multicomponent reactions. It used for synthesis of chiral α-amino nitriles. This reaction was carried out in the presence of catalytic amount of layered double hydroxides (LDHs). In this study, simple and practical method for the synthesis of this class of catalysts is provided. The results shown that LDH is the good heterogen catalyst for synthesis of chiral α-amino nitriles.

Keywords: Layered double hydroxide (LDHs), Strecker reaction, α-Amino acids, Diastereoselectivity

INTRODUCTION

To use multicomponent reactions, is a good way to synthesis many components. Application of these reactions because of the simplicity, compatibility with the environment, reducing the synthesis steps, the less chemical waste and so on has always been [1]. AccordingtoTietze, thesereactionshavehigh bond forming efficiency [2]. So manychemistsmake use of these reaction to produce various products.

One of the oldest and mostwidely use multi component reactions is Strecker that reported by Strecker for synthesis of α-amino acids in 1850. Accurse reaction between aldehyde and amine in the first step to form the imine followed by the addition of hydrogencyanidetoform α-aminonitriles as an intermediate necessary for synthesis of α-amino acids [3]. The highly used compounds in these reactions, they usually try to improve the reaction efficiency by charging the reaction conditions, such as solvent, temperature, catalyst concentration and type of the reagent and formation of side products are minimized. Among the various catalysts that have beenused so farlikeLewis acidssuch as chlorides [4,5], transition metals such as Indium [6], Rhodium [7], Ruthenium [5], Cerium [8], Nickel [4], Palladium [9], *etc.* oxides such as Aluminum, Silicon, Titanium and Manganese [10], complex compounds such as montmorillonite-KSF [11], phosphotungstic acid [12] and nanocatalyst [13] and magnetic catalysts [14] can be mentioned. Using lewis acid catalysts containing chiral ligands or chiral organic compounds, pure enantiomer α-amino acids have been synthesized [15-17].

However, this reaction due to the formation of new chiral center is also take into consideration. For selective synthesis of chiral center, chiral primary substance, chiral media or catalysts helping chiral center can be used according to the reaction mechanism which goes through *via* attaching of cyanide anion to imine bond, if the media isionic the possibility of selective formation of an enantiomer will enhance.

Layered Double Hydroxide/(LDH) are a class of compounds that are formed by repetition of layers with positive ion and also the zone between a layer containing negative ions and solvent molecules that cause neutralization. More studies of these class of compounds has been done with di- and tri-valentcations. These classes of compounds have the general formula

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$[M^{2+}_{1-x}M^{3+}_{x}(\text{-OH})_2]^{x+}[(Ann^-)_{x/n}.yH_2O]^{x-}$. Their anion can be organic or inorganic. However, these compounds many include M^+ and M^{4+} cations but these are limited [18-20]. Different applications including drug delivery [21] the hosts foranion exchange [22], absorbent of CO_2 in precursors [23-25] and catalytic role [26] for this class of compounds have been repeated.

Mixed oxides obtained by calcining LDH are used as a solid catalysts in some chemical reactions, such as aldole reaction of aldehydes and ketones, methylation, synthesis of hydrocarbons and *etc.* [27]. In these reactions the efficiency of reactions is improved usually by changing the reaction conditions such as solvent, temperature, catalyst concentration and type of reagent and the formation of side product is minimized subsequently.

EXPERIMENTAL

All the substances used in reaction are purchased from Fluka and Merck companies. To identify the products formed by ^{1}H NMR and ^{13}C NMR spectra taken by Bruker DRX 300 Avance spectrometers, respectively at 300 and 75 MHz and are used in $CDCl_3$ solvent improving reaction condition. For separation of catalyst are usedfromEppendorf centrifuge 5417R.

Synthesis of Mg-Al-Cu-NO_3 LDHs

Magnesium hydroxide (5.80 g, 0.100 mol) was added to aluminum hydroxide (1.30 g, 0.017 mol) and milled for 1 h in room temperature using a planetary ball mill. The mixture was removed in Teflon-lined stainless-steel autoclave containing 30 ml of 0.078 M $Cu(NO_3)_2.3H_2O$ solution. Then, it was heated in an oven and treated hydrothermally at 80 °C. The mixture was centrifuged. Product was washed with water and dried.

X-Ray diffraction (XRD) patterns of the prepared samples were recorded on a D/max-rA model diffractometer with Cu Karadiation in the 2θ range of 10-70 with a scanning rate of 0.08° min^{-1}. FT-IR spectra of the samples were collected suing KBr pellets on a Vector 22 Fourier transformation infrared spectroscope in reflectance mode, at the range of 400-4000 cm^{-1}, with a resolution of 2 cm^{-1}. Typicalpowder XRD patterns of the Mg-Al-Cu-LDH isshown in Fig. 1.

The characteristic diffraction peaks of the LDH-phase could be clearly observed which is in good agreement with that reported in the literature [28]. According to the JCPDS 37-0630, the characteristic diffractions around 2θ 11.4°, 22.6°, 34.5°, 38.7°, 45°, 60.3° and 61.5° can be assigned to the typical (003), (006), (012), (015), (018), (110) and (113) face of Mg-containing Cu-Al-LDH.

Figure 2 shows the FT-IR spectra of the Mg-Al-Cu-LDH. The strong and broad band centered on 3450 cm^{-1} was assigned to the OH stretching vibrations of the hydroxyl groups in the LDH layers and the interlayer water molecules, and another absorption band corresponding to water deformation was recorded around 1639 cm^{-1}. The absorption peak at 1365 cm^{-1} (CO_3^{2-}) showed that the LDH products contain some CO_3^{2-} [29], whereas the response around 1500 cm^{-1} may be assigned to the C-C site. The peaks from 400 to 1000 cm^{-1} were attributed to the stretching and bending vibrations of M-O and M-OH [30].

General Procedure for the Synthesis of Methylated Amino Acids

1 mmol amino acid was mixed with 3 ml methanol in balloon. The balloon was placed in a bath of water and acetone until the temperature of balloon contents reached to -5 °C, then 3 mmol thionyl chloride was slowly added to reaction vessel. After thionyl chloride is increased, all the amino acids become soluble and the environment pH decreased to 1. The reaction mixture is being mixed at room temperature for 5 h and the progress of the reaction using TLC with a mixture of H_2O:MeOH:EtOH (10:2:1) and the detector ninhydrine followed. After completion of the reaction, methanol was evaporated by hot water bath, and solid product was dried. In the following, for purification, the resulting precipitate was dissolved in the minimum amount of methanol and diethylether was added as antisolvent finally, pure precipitate obtained was filtered.

General Procedure for the Synthesis of α-Amino Nitriles (4a-h)

mmol resulting methylated amino acid was dried and dissolved with 5 ml methanol. 20 mg catalyst was added to reaction vessel and after being mixed for 5 min. 1 mmol benzaldehyde was added to the balloon content reaction progress was followed by TLC (hexane:etylacetate

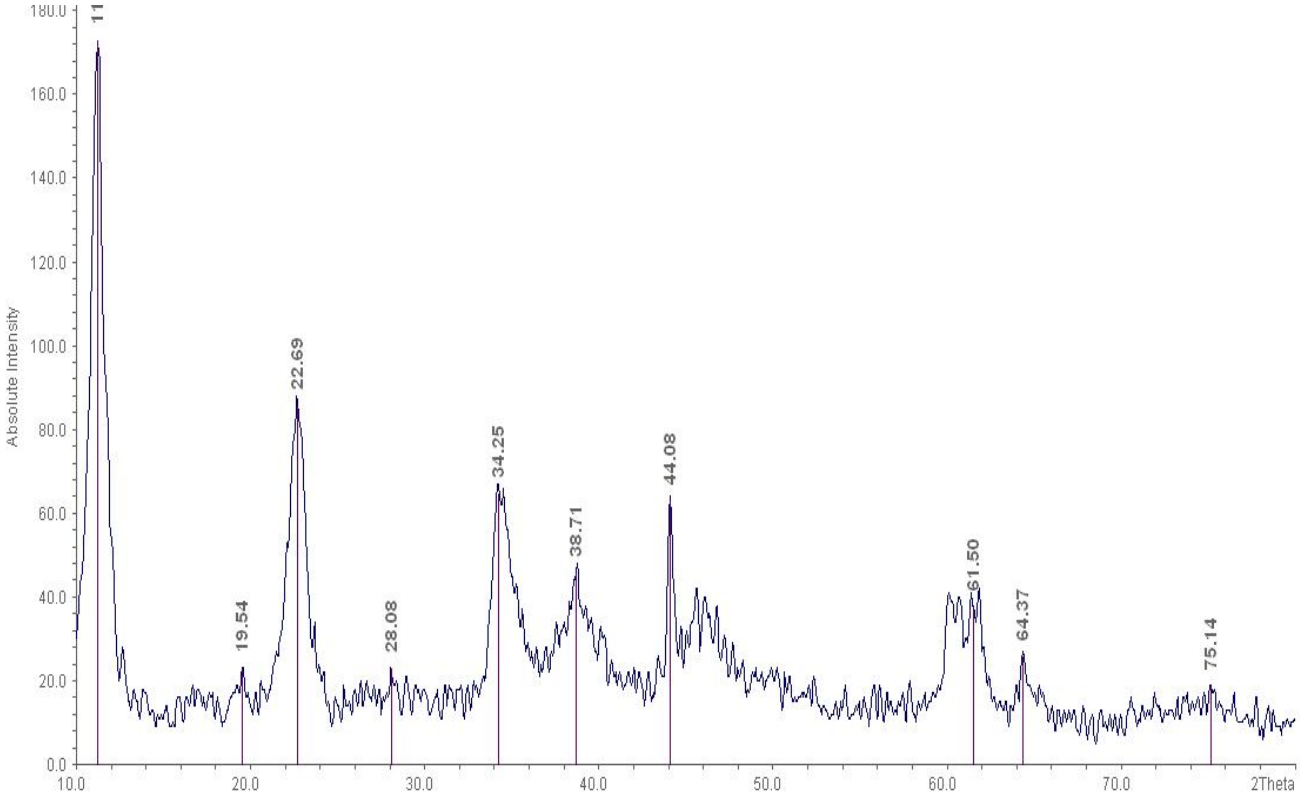


Fig. 1. XRD patterns of Mg-Al-Cu-LDH.

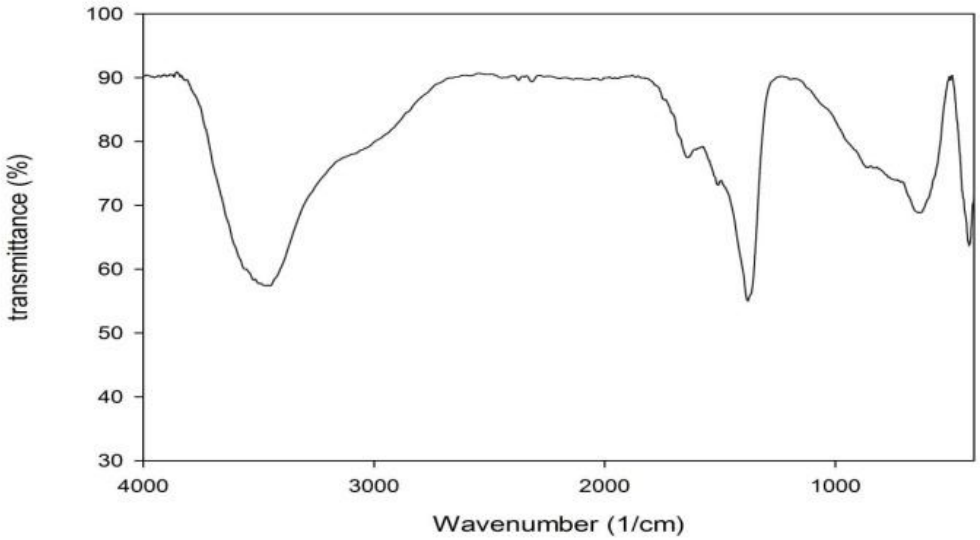
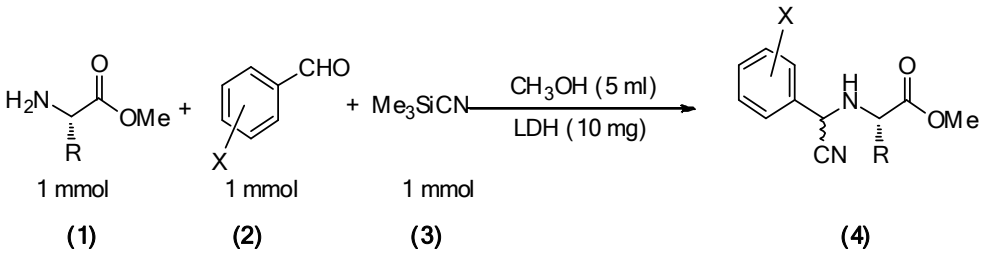


Fig. 2. IR Spectra of Mg-Al-Cu-LDH.



*Scheme 1.*General reaction of preparation amino nitrils in optimized condition

3:1) after formation of imine, 1 mmol trimethyl silyl cyanide was added (Scheme 1). Reaction progress was followed and after 3 h, the contents of the balloon were transferred to the special centrifuges container (room temperature, 3000 rpm, 10 min) and the catalyst was removed by decantation.

The remaining solution was purifiedby plate chromatography and the product was isolated.

Spectral Data for Compounds

Methyl-2-((cyano(p-tolyl)methyl)amino)-3-(1H-indol-3-yl)propanoate (4a). (mixture of two diastereomers (58:42)). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.35 (s, 6H, Me, mixture of two diastereomers), 3.20 (dd, 2H, J = 11.7, 4.16, CH_2, mixture of two diastereomers), 3.29-3.30 (m, 2H, CH_2, mixture of two diastereomers), 3.68 (s, 3H, -OMe, diastereomer A), 3.74 (s, 3H, -OMe, diastereomer B), 3.97-4.01 (m, 1H, CHα, diastereomer A), 4.24-4.26 (m, 1H, CHα, diastereomer B), 4.61 (s, 1H, CHCN, diastereomer A), 4.82 (s, 1H, CHCN, diastereomer B), 7.01 (d, 1H, J = 2.1, 2H, CH Ar, diastereomer A), 7.05 (d, 1H, J = 2.0, 2H, CH Ar, diastereomer B), 7.12-7.20 (m, 4H, CH Ar, mixture of two diastereomers), 7.21 (s, 1H, =CH indole, diastereomer A), 7.26 (s, 1H, =CH indole, diastereomer B), 7.32 (d, 2H, J = 8.0, CH Ar, mixture of two diastereomers), 7.53 (t, 2H, J = 8.2, CH Ar, mixture of two diastereomers), 7.60 (d, 2H, J = 7.3, CH Ar, mixture of two diastereomers), 7.84 (d, 1H, J = 1.3, CH Ar, diastereomer A), 7.85-7.87 (m, 1H, CH Ar, diatsereomer B).

^{13}C NMR (75 MHz, $CDCl_3$) δ 29.1, 29.2, 31.4, 31.9, 52.1, 52.2, 52.8, 52.9, 54.6, 59.0, 59.4, 110.1, 110.4, 111.3, 118.6, 118.8, 118.9, 119.5, 121.9, 122.1, 122.9, 123.1, 127.4, 128.3, 129.5, 129.6, 130.1, 131.4, 131.5, 136.1, 136.2, 139.1, 147.2, 174.0, 174.1.

Methyl-2-((cyano(phenyl)methyl)amino)-3-(1H-indol-3-yl) propanoate (4b). (mixture of two diastereomers (56:44)). ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.20-3.29 (m, 2H, CH_2 mixture of two diastereomers), 3.30-3.34 (m, 2H, CH_2 mixture of two diastereomers), 3.68 (s, 3H, -OMe, diastereomer A), 3.82 (s, 3H, -OMe, diastereomer B), 4.00 (dd, 1H, J = 7.5, 5.0, CHα, diastereomer A), 4.31 (dd, 1H, J = 8.5, 5.0, CHα, diastereomer B), 4.65 (s, 1H, CHCN, diastereomer A), 4.86 (s, 1H, CHCN, diastereomer B), 6.90 (d, 1H, J = 2.20, CH Ar, diasteromer A), 7.05 (dd, 1H, J = 11.4, 2.28, CH Ar, diastereomer B), 7.15-7.21 (m, 6H, CH Ar, mixture of two diastereomers), 7.22 (s, 1H, CH Arindole, diastereomer A), 7.36 (s, 1H, CH Arindole, diastereomer B), 7.40-7.44 (m, 4H, CH Ar, mixture of two diastereomers), 7.57-7.62 (m, 4H, CH Ar, mixture of two diastereomers), 7.68 (dd, 2H, J = 8.0,1.5, CH Ar, mixture of two diastereomers).

^{13}C NMR (75 MHz, $CDCl_3$) δ29.3, 29.7, 52.2, 53.0, 53.1, 53.2, 54.9, 58.6, 59.1, 59.5, 108.8, 110.0, 111.0, 111.3,118.5, 118.6, 118.7, 119.5, 121.9, 122.1, 122.9, 123.1, 128.4, 128.5, 128.9, 129.0, 129.1, 134.4, 136.1, 140.7, 173.3, 173.9, 174.0, 174.1.

Methyl-2-((cyano(3-nitrophenyl)methyl)amino)-3-(1H-indol-3-yl) propanoate (4c). (mixture of two diastereomers (52:48)). ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.12-3.19 (m, 2H, CH_2 mixture of two diastereomers), 3.23-3.29 (m, 2H, CH_2 mixture of two diastereomers), 3.72 (s, 3H, -OMe, diastereomer A), 3.83 (s, 3H, -OMe, diastereomer B), 3.92-3.96 (m, 1H, CHα, diastereomer A), 4.12 (q, 1H, J = 7.1, CHα, diastereomer B), 5.37 (s, 1H, CHCN, diastereomer A), 5.51 (s, 1H, CHCN, diastereomer B), 7.11-7.13 (m, 1H, CH Arindole, diasteromer A), 7.48 (d, 1H, J = 4.5, CH Ar, diastereomer A), 7.51 (d, 1H, J = 4.5, CH

Ar, diastereomer B), 7.57 (d, 2H, *J* = 5.9, CH Arindole, diastereomer A), 7.62 (d, 2H, *J* = 6.7, CH Ar, diastereomer B), 8.11-8.17 (m, 2H,CH Ar, mixture of two diastereomers), 8.19-8.21 (m, 2H, CH Ar, mixture of two diastereomers).

^{13}C NMR (75 MHz, $CDCl_3$) δ 29.3, 297, 52.6, 54.1, 56.6, 58.0, 58.5, 60.4, 108.7, 109.5, 111.0, 118.4, 119.7, 119.8, 122.2, 122.3, 123.0, 123.1, 123.3, 123.6, 126.8, 126.9, 129.9, 131.8, 133.1, 134.9, 136.3, 136.4, 143.2, 144.4, 172.9, 173.8, 174.0.

Methyl-2-((cyano(*p*-tolyl)methyl)amino)-3-(4-hydroxyphenyl) propanoate (4d). (mixture of two diastereomers (53:47)). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.34 (s, 3H, Me, diastereomer A), 2.36 (s, 3H, Me, diastereomer B), 2.86 (dd, 2H, *J* = 14, 7.3, CH_2, mixture of two diastereomers), 2.95-3.10 (m, 2H, CH_2, mixt,ure of two diastereomers), 3.80 (dd, 1H, *J* = 7.8, 5.7, CHα, diastereomer A), 3.69 (s, 3H, -OMe, diastereomer A), 3.70 (s, 3H, -OMe, diastereomer B), 4.11 (dd, 1H, *J* = 8.8, 5.2, CHα, diastereomer B), 4.55 (s, 1H, CHCN, diastereomer A), 4.79 (s, 1H, CHCN, diastereomer B), 6.62 (d, 1H, *J* = 8.3, CH Ar, diastereomer A), 6.67 (t, 2H, *J* = 8.7, CH Ar, mixture of two diastereomers), 6.99 (dd, 1H, *J* = 8.5, 2.8, CH Ar, diastereomer B), 7.03 (d, 1H, *J* = 8.4, CH Ar, diastereomer A), 7.18 (t, 3H, *J* = 4.6, CH Ar, mixture of two diastereomers), 7.27 (t, 2H, *J* = 5.9, CH Ar, mixture of two diastereomers), 7.60 (d, 1H, *J* = 5.2, CH Ar, diastereomer B).

^{13}C NMR (75 MHz, $CDCl_3$) δ 21.5, 22.7, 28.8, 29.7, 38.5, 38.8, 52.2, 52.7, 52.9, 59.8, 115.3, 115.5, 115.6, 115.7, 127.3, 127.6, 128.2, 128.5, 128.9, 129.3, 129.5, 129.6, 131.2, 131.3, 139.1, 141.6, 154.6, 155.1, 172.5, 173.8.

Methyl-2-((cyano(phenyl)methyl)amino)-3-(4-hydroxyphenyl) propanoate (4e). (mixture of two diastereomers (55:45)). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.81-2.89 (m, 1H, CH_2, mixture of two diastereomers), 2.98-3.11 (m, 2H, CH_2, mixture of two diastereomers), 3.29 (dd, 1H, *J* = 13.6, 5.1, CHα, diasterreomer A), 3.75 (s, 6H, -OMe, mixture of two diastereomers), 4.14 (dd, 1H, *J* = 8.8,5.1 Hz, CHα, diasterreomer B), 4.59 (s, 1H, CHCN, diastereomer A), 4.83 (s, 1H, CHCN, diastereomer B), 6.67 (d, 2H, *J* = 6.3, CH Ar, diastereomer A), 6.70 (d, 2H, *J* = 5.9, CH Ar, diastereomer B), 6.75 (d, 2H, *J* = 9.4, CH Ar, mixture of two diastereoers), 6.97 (dd, 2H, *J* = 6.0, 2.1, CH Ar, mixture of two diastereomers), 7.02 (t, 2H, *J* = 2.6, CH Ar, CH Ar, mixture of two diastereomers), 7.05 (brs, 1H, CH Ar, diastereomer A), 7.34-7.42 (m, 6H, CH Ar, mixture of two diastereomers), 7.68 (dd, 2H, *J* = 8.0,1.6, CH Ar, mixture of two diastereomers).

^{13}C NMR (75 MHz, $CDCl_3$) δ 29.7, 30.1, 38.5, 38.9, 52.3, 52.9, 59.8, 60.5, 115.3, 115.5, 115.6, 115.7, 127.4, 127.6, 128.1, 128.5, 128.7, 129.2, 130.3, 130.4, 130.8, 1331.2, 134.1, 134.2, 154.7, 155.2, 172.4, 173.8.

Methyl-2-((cyano(3-nitrophenyl)methyl)amino)-3-(4-hydroxyphenyl) propanoate (4f). (mixture of two diastereomers (63:37)). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.78-2.85 (m, 1H, CH_2, one of diastereomers), 2.99-3.07 (m, 2H, CH_2, mixt,ure of two diastereomers), 3.27 (dd, 1H, *J* = 5.1, 4.7, CH_2, one of diastereomers), 3.70 (s, 3H, -OMe, diastereomer A), 3.77 (s, 3H, -OMe, diastereomer B), 4.12 (q, 1H, *J* = 7.2, CHα, diasterreomer A), 4.19 (dd, 1H, *J* = 9.0, 4.8, CHα, diasterreomer B), 4.62 (s, 1H, CHCN, diastereomer A), 4.99 (s, 1H, CHCN, diastereomer B), 6.69 (dd, 3H, *J* = 8.5, 2.7, CH Ar, mixtue of two diastereomers), 6.77 (d, 1H, *J* = 8.5, CH Ar, diastereomer A), 6.95 (td, 3H, *J* = 8.8, 2.4, CH Ar, mixtue of two diastereomers), 7.04 (d, 1H, *J* = 8.5, CH Ar, diastereomer B), 7.54 (t, 2H, *J* = 8.1, CH Ar, mixture of two diastereomers), 7.72-7.74 (m, 1H, diastereomer A), 7.94 (s, 1H, CH Ar, diastereomer B), 8.01 (dt, 1H, *J* = 7.7, 1.1, CH Ar, diastereomer A), 8.18-8.24 (m, 3H, CH Ar, mixture of two diastereomers), 8.47 (t, 1H, *J* = 1.7, CH Ar, diastereomer A).

^{13}C NMR (75 MHz, $CDCl_3$) δ 29.6, 38.5, 38.8, 52.5, 52.6, 55.1, 59.5, 60.6 115.4, 115.6, 115.7, 117.4, 122.7, 127.4, 128.0, 128.4, 129.6, 130.2, 130.3, 130.4, 130.8, 133.9, 136.4, 136.9, 154.8, 155.1, 173.6, 173.8.

Methyl-2-((cyano(phenyl)methyl)amino)-3-phenyl-propanoate (4g). (mixture of two diastereomers (71:29)). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.88-2.97 (m, 2H, CH_2, mixture of two diastereomers), 3.10-3.19 (m, 2H, CH_2, mixture of two diastereomers), 3.75 (s, 3H, -OMe, diastereomer A), 3.78 (s, 3H, -OMe, diastereomer B), 3.86 (dd, 1H, *J* = 7.7, 5.5, CHα, diastereomer A), 3.99 (d, 1H, *J* = 5.8, CHα, diastereomer B), 4.55 (s, 1H, CHCN, diastereomer A), 4.84 (s, 1H, CHCN, diastereomer B), 7.11-7.28 (m, 4H, CH Ar, mixture of two diastereomers), 7.30-7.32 (m, 2H, CH Ar, mixture of two diastereomers), 7.36-7.39 (m, 2H, CH Ar, mixture of two diastereomers), 7.40 (dd, 1H, *J* = 7.7, 5.1, CH Ar, mixture of two diastereomers),

7.49-7.62 (m, 3H, CH Ar, mixture of two diastereomers), 7.68-7.75 (m, 2H, CH Ar, mixture of two diastereomers), 7.82 (dd, 2H, J = 8.3 Hz, J = 1.2, CH Ar, mixture of two diastereomers),

^{13}C NMR (75 MHz, $CDCl_3$) δ 39.4, 39.7, 52.2, 52.9, 53.0, 53.3, 59.6, 60.4, 110.1, 116.7, 117.0, 118.4, 118.6, 127.0, 127.1, 127.4,127.5,128.5, 128.6, 128.7, 128.8, 128.9,129.0, 129.1, 129.3, 129.4, 129.7, 131.6, 132.8,132.9, 133.2, 135.6, 136.1, 136.4, 136.8, 173.5, 173.6.

Methyl-2-((cyano(3-nitrophenyl)methyl)amino)-3-phenylpropanoate (4h). (mixture of two diastereomers (61:39)). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.83-2.96 (m, 2H, CH_2, mixture of two diastereomers), 3.10-3.19 (m, 2H, CH_2, mixture of two diastereomers), 3.41 (dd, 1H, J = 13.5, 4.8, CHα, diastereomer A), 3.71 (s, 3H, -OMe, diastereomer A), 3.75 (s, 3H, -OMe, diastereomer B), 4.23 (dd, 1H, J = 9.2, 4.7, CHα, diastereomer B), 4.70 (s, 1H, CHCN, diastereomer A), 4.99 (s, 1H, CHCN, diastereomer B), 7.16 (td, 2H, J = 13.5, 2.5, CH Ar, mixture of two diastereomers), 7.23-7.30 (m, 3H, CH Ar, mixture of two diastereomers), 7.53 (dd, 1H, J = 7.9, 2.8, CH Ar, diastereomer A), 7.59 (d, 1H, J = 7.6, CH Ar, diastereomer B), 7.72 (td, 1H, J = 6.8,0.7 Hz, CH Ar, diastereomer A), 8.04 (d, 1H, J = 7.7 Hz, CH Ar, diastereomer B), 8.19 (d, 1H, J = 8.2, CH Ar, diastereomer A), 8.24 (dd,1H, J = 4.3, 1.7, CH Ar, diastereomer B), 8.49 (t, 1H, J = 1.8, CH Ar, diastereomer A).

^{13}C NMR (75 MHz, $CDCl_3$) δ 39.5, 39.7, 52.1, 52.2, 52.3, 52.6, 59.4, 60.5, 118.0, 118.3, 127.0, 127.1, 128.4, 128.6, 128.7, 128.8, 128.9, 129.1, 129.2, 129.4, 129.6, 129.7,132.7, 132.8, 135.1, 135.2,136.1, 136.7, 173.5, 173.6.

RESULTS AND DISCUSSION

Initially, to determine the optimum conditions of synthesis, reaction of methylated phenylalanine with benzaldehyde was selected as a model and reaction parameters were studied (Scheme 2). To do this, the effect of catalyst used in synthesis of derivative was evaluated that the catalyst LDH was selected as the desired catalyst and then the effects of solvent was studied after that the effect of the catalyst used was evaluated.

It should be noted that the reaction of methylation of the amino acids are done in accordance with industrial protocols and require no optimization.

Table 1 shows the percentage yields of products in presence 20 mg different catalysts. As can be seen reaction in presence of LDH has the highest yield.

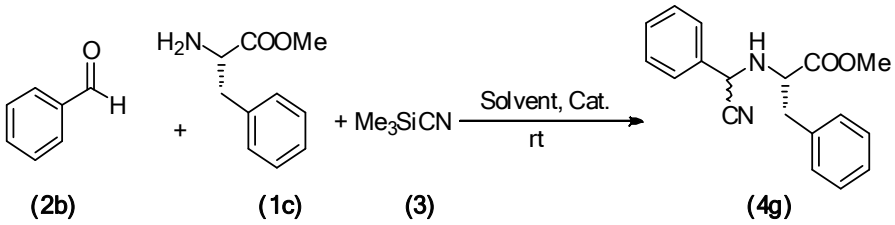
After determining LDH catalyst as the best catalyst, in order to optimized the reaction solvent, protic polar solvents such as methanol and aprotic solvent such as dichloromethane and also a solvent such as acetonitrile and solvent free condition were used. The final results are given in Table 2.

First, 1 mmol methylated phenylalanine and 5 ml solvent, followed by 20 mg catalyst were mixed together, after 5 min, 1 mmolbenzaldehyde was added to the contents of the flask. After 30 min, the reaction progress was followed by TLC. When the imine structure was confirmed, the value of 1 mmol trimethylsilylcyanide was added and TLC taken again.

TLC showed that imine formation in acetonitrile is harder and slower. Methylated amino acid was dissolved in methanol better. Progress was followed by TLC. The results showed that the best imines were formed in methanol and dichloromethane. It should be noted that in term of solvent free,gridding materials in crucible was used to raise the chances of molecules colliding. In solvent free, reaction was progress but slow. The results show that methanol was selected as the optimal solvent.

After determining methanol as a solvent, amount of catalyst used in reaction at room temperature was evaluated. The intended reaction (methylated phenyl alanine reaction with benzaldehyde) was done with different levels of LDH catalyst, after 30 min, the intensity of the spots on the TLC showed that all of them have produced imine at the same ratio. 1 mmol trimethylsilylcyanide was added and reaction progress was followed by the TLC. (As there has been no molecular structure set for this catalyst, molar mass and M percent of catalyst consumption can't be reported) (Table 3).

After optimization of reaction condition, derivatives of α-amino nitriles were synthesized and diastereoselectivity of new chiral center were determined by the comparison of the integral of singlet peaks in σ = 4-5.5 ppm (CHCN) (Table 4).



Scheme 2. The model reaction

Table 1. Effect of Catalyst for the Model Reaction

Catalyst	Yield (%)
-	0
$H_3[P(W_3O_{10})_4].H_2O$	80
(s)- (+)-1,1′-Binaphtyl-2-2′diyl hydrogen phosphate	39
S-Proline	78
LDH	82

Table 2. Effect of Solvent for the Model Reaction

Entry	Solvent	Yield (%)
1	-	54
2	Methanol	84
3	Dichloromethan	82
4	Acetonitril	76

Table 3. Effect of amount of LDH for the Model Reaction

Entry	Catalyst (mg)	Yield (%)
1	5	24
2	10	59
3	20	85
4	40	63

CONCLUSIONS

The results show that in presence of catalytic amount of Layered double hydroxides (LDH), the strecker reaction proceeds selectively. This can be due the ionic environment. It is also concluded that selectivity increases when using smaller side chain amino acids.

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