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14.
**BASIC HYDROTALCITE CATALYZED KNOEVENAGEL
CONDENSATION: SYNTHESIS OF 3-METHYL-4-
[(1,3-DIPHENYL-1H-PYRAZOL-4-YL)-METHYLENE]-1-
PHENYLPYRAZOLIN-5-(4H)-ONES**

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Abstract:

Basic hydrotalcites catalyzed ecofriendly, convenient and excellent yielding (80-89%) synthesis of 3-Methyl-4-[(1,3-diphenyl-1H-pyrazol-4-yl)-methylene]-1-phenylpyrazolin-5-(4H)-ones (**3a-e**) has been carried out by the Knoevenagel condensation reaction of 1,3-diphenyl-1H-pyrazole-3-carbaldehydes (**1a-e**) and 3-methyl-1-phenylpyrazolin-5-(4H)-one. Mild reaction conditions, reusable and user friendly catalyst creates affirmative effect on nature. Recycling effectiveness of the catalyst for three cycles remained constant; which confirmed by the reaction completion time and the product yield (entry **3e**, 84%). Solvent of reactions were recovered, and used for the reaction after distillation for obtaining consistent result.

Keywords:

Basic hydrotalcites, Knoevenagel condensation, pyrazolinone, pyrazoles, ecofriendly.

Introduction:

Application of solid base catalysts have been of large interest due to their tunable basicity, environmentally acceptable nature, and capacity to catalyze diverse reactions.^{1,2} This approach has attracted intense interest and has been reviewed extensively.^{2,3} In organic synthesis one of the basic carbon-carbon bond formation reactions is the Knoevenagel condensation reaction. These condensation reactions require acid or base catalyst and prolonged heating. Present growing concerns about controlling important, and protecting future resources, design of routes having environmentally benign characteristics have attracted considerable interest in organic synthesis⁴. An ideal synthesis should produce the expected products in high yields with the help of catalyst possessing the properties such as effectiveness, recyclability, ease in handling and its minimum loading.

Results and discussion:

Basic hydrotalcites⁵ have often been discussed as promising catalyst for carrying out various condensation reactions^{4,6}. Here in report the condensation of 1,3-diphenyl-1H-pyrazole-3-carbaldehyde with 3-methyl-1-phenylpyrazolin-5-(4H)-one for obtaining 3-methyl-4-[(1,3-diphenyl-1H-pyrazol-4-yl)-methylene]-1-phenylpyrazolin-5-(4H)-ones in an excellent yields. All the reactions were carried out at room temperature on stirring for 25-30 minutes (Table 1). In particular, the solvent 1,4-Dioxane was used, which is non-chlorinated and polar enough to facilitate the reactions. The basic hydrotalcite catalyst was used and which is recyclable catalyst. For the catalyst separation and product re-crystallization from the reaction mixture; the reaction mixture was directly heated and filtered. The residue was washed with hot 1,4-dioxane. Combined filtrate and washings were kept overnight at room temperature. Obtained crystals were filtered, dried and weighed to accomplish the products in excellent yields. During this re-crystallization process catalyst got separated. This separated catalyst was reused for carrying out the same reactions. The efficiency of catalyst