

List of papers presented at Conference / Symposia

1. **N. Narkhede** and A. Patel, 12-Tungstophosphoric acid anchored on to Zeolite H β : Synthesis, Characterization and use as catalyst for Solvent Free Biginelli Reaction. Regional Science Congress on 'Science for shaping future of India', Department of Chemistry, The M. S. University, September 15th-16th, 2012.
2. **N. Narkhede** and A. Patel, Synthesis of solid acid catalysts comprising Zeolite Beta and Heteropoly acids: Synthesis, Characterization and Biodiesel production by esterification of Free Fatty Acids. 4th Interdisciplinary symposium on Materials Chemistry, BARC, December 11-15, 2012.
3. **N. Narkhede**, S. Singh and A. Patel, Solvent Free Aerobic Oxidation of Alkenes and alcohols using undecamolybdophosphate anchored MCM-41. 21st National symposium on catalysis, Catalysis for Sustainable Development, (CATSYMP-21), IICT-Hyderabad, February 11-13, 2013.
4. **N. Narkhede** and A. Patel, Biodiesel synthesis by esterification of Palmitic acid over 12-Tungstophosphoric acid anchored to Zeolite HBeta. National Symposium on Modern Trends in Chemistry, Department of Chemistry, Faculty of Science, The M S. University of Baroda, 21-23, March, 2013.

Papers published from work excluding thesis

1. A. Patel and **N. Narkhede**, 12-tungstophosphoric acid anchored to zeolites H β : Synthesis, characterization and biodiesel production by esterification of oleic acid with methanol, *Energy Fuels*, 26, 6025, (2012).
2. **N. Narkhede**, A. Patel, Greener, solvent free three component Biginelli reaction over different recyclable solid acid catalysts. *J. Porous Mater.*, 21, 579, (2014).
3. **N. Narkhede**, A. Patel and S. Singh, Mono lacunary phosphomolybdate supported to MCM-41: Synthesis, characterization and solvent free aerobic oxidation of alkenes and alcohols, *Dalton Trans.*, 43, 2512, (2014).
4. **N. Narkhede**, V. Brahmkhatri, A. Patel, Efficient Synthesis of Biodiesel From Waste Cooking Oil Using Solid Acid Catalyst Comprising 12-Tungstosilicic Acid and SBA-15. *Fuel*, 135, 253, (2014).
5. S. Singh, **N. Narkhede** and A. Patel, Aerobic oxidation of alcohols and alkenes over a novel lacunary phosphomolybdate anchored to zeolite H β , *RSC Adv.*, 5, 36270, (2015).

PAPER

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Biodiesel synthesis *via* esterification and transesterification over a new heterogeneous catalyst comprising lacunary silicotungstate and MCM-41

Anjali Patel* and Nilesh Narkhede

Mono-vacant silicotungstate anchored to MCM-41 was synthesized by a direct post-synthesis grafting method and characterized by BET surface area measurements, FT-IR, ^{29}Si MAS NMR, XRD and TEM. The catalytic application of the material was established for acid catalyzed synthesis of biodiesel *via* esterification of oleic acid and transesterification of different edible and non-edible oils such as waste cooking oil, jatropha oil, sunflower oil, cotton seed oil and mustard oil. A kinetic study was carried out for the esterification of oleic acid and it was found that esterification of oleic acid follows first order kinetics with the calculated activation energy $E_a = 49.8 \text{ kJ mol}^{-1}$ and pre-exponential factor $A = 8.7 \times 10^4 \text{ min}^{-1}$. The expected mechanism for the biodiesel synthesis *via* esterification and transesterification has also been proposed.

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Green Chemistry



CRITICAL REVIEW

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Recent progress on supported polyoxometalates for biodiesel synthesis *via* esterification and transesterification

Nilesh Narkhede, Sukriti Singh and Anjali Patel*

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Biodiesel is now recognized as a "green fuel" that has several advantages over conventional diesel. In the present review we discuss the catalytic esterification and transesterification reactions to the clean synthesis of biodiesel, over most readily investigated supported polyoxometalates to meet future societal demands. Here for the first time, we are reviewing biodiesel synthesis using supported lacunary polyoxometalates; also how the prevailing reaction conditions like reaction time, alcohol content, temperature, and catalyst amount affect the catalytic activity of the catalyst is discussed in detail. The new results for supported lacunary polyoxometalates are included where the effect of catalysts and supports was correlated with the catalytic activity.



RSC Advances

PAPER

Room temperature acetalization of glycerol to cyclic acetals over anchored silicotungstates under solvent free conditions

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Nilesh Narkhede and Anjali Patel*

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Heterogeneous catalysts comprising of parent Keggin type silicotungstate as well as monolacunary silicotungstate anchored to MCM-41 were synthesized and characterized by several physicochemical methods. A solvent free green route towards valorisation of glycerol *via* acetalization with benzaldehyde has been proposed. Both the catalysts showed very good activity as well as selectivity towards dioxolane derivatives within a short reaction time and at room temperature. The tuning of the acidity of the parent silicotungstate leads to an increase in the selectivity towards 1,3-dioxolane. The catalysts were also recycled up to four times without any significant loss in the conversion. The excellent performance of these mesoporous catalysts is attributed to their combination of acidity, wide pores and large specific surface area.



RSC Advances

PAPER



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Facile synthesis of glycerol carbonate *via* glycerolysis of urea catalysed by silicotungstates impregnated into MCM-41

Nilesh Narkhede and Anjali Patel*

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The present contribution includes the solvent free environmentally benign route towards synthesis of glycerol carbonate *via* glycerolysis of urea. The parent as well as mono lacunary silicotungstates impregnated into MCM-41 were used as efficient catalysts. The effect of different reaction parameters on the conversion of glycerol was investigated in order to optimize the reaction parameters for maximum conversion. The activities of both the catalysts have been correlated with the structural features and acidity of the catalysts and the possible mechanism has also been proposed. The catalysts were also recycled up to four times without significant loss in the activity.

Biodiesel Production by Esterification of Oleic Acid and Transesterification of Soybean Oil Using a New Solid Acid Catalyst Comprising 12-Tungstosilicic Acid and Zeolite H β

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S Supporting Information

ABSTRACT: A series of catalysts containing 10–40 wt % of 12-tungstosilicic acid anchored to zeolite H β were synthesized and characterized by different physicochemical techniques. Their catalytic activity was evaluated for esterification of free fatty acid and oleic acid as well as transesterification of soybean oil with methanol. The effects of reaction variables such as catalyst loading, methanol to acid ratio, reaction time, and temperature on the conversion were studied. The kinetic study was carried out for esterification of oleic acid, and its Arrhenius constant (A) and activation energy (E_a) were evaluated. The maximum conversion obtained for esterification of oleic acid and transesterification of soybean oil were 86% and 95%, respectively. Also the catalyst was recycled up to four cycles without any loss in the conversion.

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Cite this: *RSC Adv.*, 2014, 4, 64379

Efficient synthesis of biodiesel over a recyclable catalyst comprising a monolacunary silicotungstate and zeolite H β

Nilesh Narkhede and Anjali Patel*

This paper presents a study of esterification of oleic acid as well as transesterification of soybean oil with methanol over a solid acid catalyst comprising a monolacunary silicotungstate anchored to zeolite H β . The optimization of the reaction parameters were carried out to get the maximum conversion. The 3.5 wt% catalyst charge at 60 °C showed 82% conversion of oleic acid with a 1 : 20 mole ratio of acid to alcohol in 10 h whereas the 4 wt% catalyst charge at 65 °C showed 96% conversion of soybean oil with a ratio of oil to alcohol of 1 : 4 (w/w) in 8 h. The catalyst was found to be readily reusable and exhibited consistency in activity upon reuse four times without appreciable loss in the conversion.

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