

Green Chemistry

Cutting-edge research for a greener sustainable future

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IN THIS ISSUE

ISSN 1463-9262 CODEN GRCHFJ 12(4) 525–728 (2010)



Cover

See Jungnickel *et al.*, pp. 593–601. Analyzing 30 ionic liquids with 13 bacterial and fungal strains we determined a surface activity dependant anti-microbial mode of action.

Image reproduced by permission of Christian Jungnickel from *Green Chemistry*, 2010, **12**, 593.



Inside cover

See Bozell and Petersen, pp. 539–554. Chemical products from carbohydrates will play a crucial role in the success of integrated biorefinery development.

Image reproduced by permission of Joseph J. Bozell from *Green Chemistry*, 2010, **12**, 539.

CRITICAL REVIEWS

539

Technology development for the production of biobased products from biorefinery carbohydrates—the US Department of Energy’s “Top 10” revisited

Joseph J. Bozell* and Gene R. Petersen

A review of recent methodology for the conversion of carbohydrates to biobased products identifies several new opportunities for the biorefinery and suggests that broad technology development will play the most important role in determining how renewable carbon is used in chemical production.

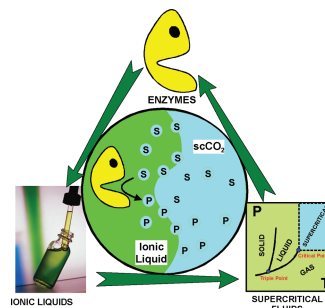


555

Enzymes in neoteric solvents: From one-phase to multiphase systems

Pedro Lozano*

Biotransformations based on neoteric solvents, such as ionic liquids (ILs), supercritical carbon dioxide ($scCO_2$) and fluorinated solvents (FSs), are interesting alternatives to organic solvents for designing clean synthetic chemical processes to obtain pure products directly. Enzyme behaviour in $scCO_2$ and ILs, as well as the phase behaviour of ILs/ $scCO_2$, are key parameters to be taken into account for carrying out integral green bioprocesses in continuous operation. Experimental approaches, reactor design and results are discussed in this Critical review.



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Green Chemistry (print: ISSN 1463-9262; electronic/ISSN 1463-9270) is published 12 times a year by the Royal Society of Chemistry, Thomas Graham House, Science Park, Milton Road, Cambridge, UK CB4 0WF.

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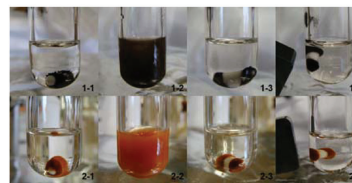
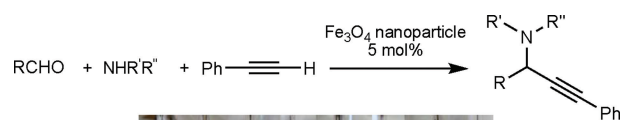
COMMUNICATIONS

570

Fe₃O₄ nanoparticles: a robust and magnetically recoverable catalyst for three-component coupling of aldehyde, alkyne and amine

Tieqiang Zeng, Wen-Wen Chen, Ciprian M. Cirtiu, Audrey Moores, Gonghua Song* and Chao-Jun Li*

A robust, safe and magnetically recoverable Fe₃O₄ nanoparticle catalyzed three-component coupling of aldehyde, alkyne, and amine (A³-coupling) was developed.

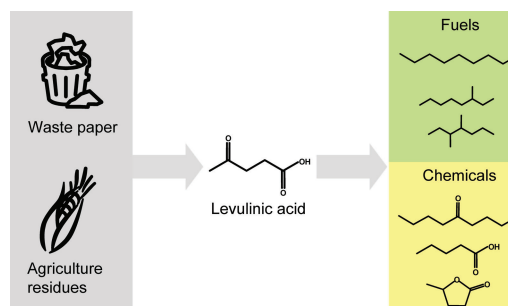


574

Catalytic upgrading of levulinic acid to 5-nonanone

Juan Carlos Serrano-Ruiz, Dong Wang and James A. Dumesic*

Levulinic acid, a biomass derivative obtained from waste and residues, can be converted to fuels and chemicals by dehydration/hydrogenation and C–C coupling reactions over Pd/Nb₂O₅. These chemicals are obtained with high yields using a limited number of reactors and separation steps.

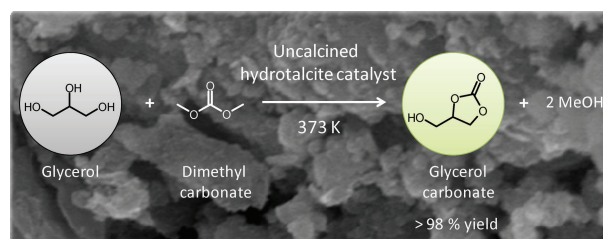


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Synthesis of glycerol carbonate from glycerol and dialkyl carbonates using hydrotalcite as a reusable heterogeneous base catalyst

Atsushi Takagaki, Ken Iwatani, Shun Nishimura and Kohki Ebitani*

An uncalcined Mg–Al hydrotalcite catalyst involving hydromagnesite with a high surface area exhibited high activity for glycerol carbonate synthesis from glycerol and dialkyl carbonates under moderate reaction conditions.

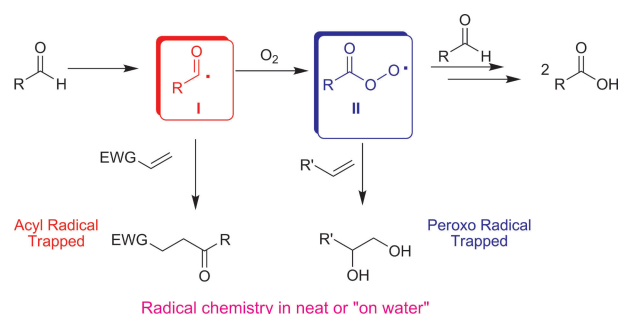


582

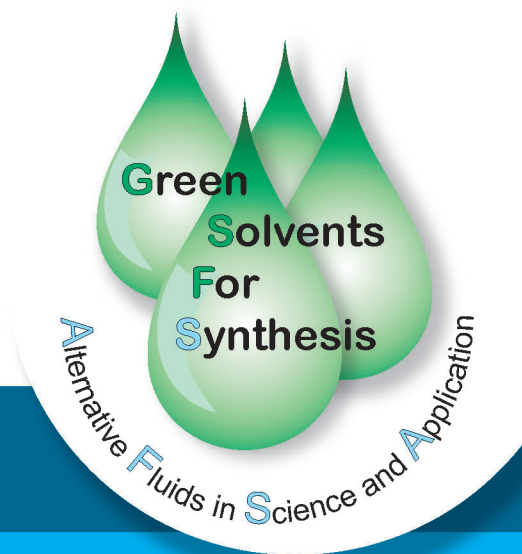
Straightforward radical organic chemistry in neat conditions and “on water”

Nelly Shapiro, Maria Kramer, Israel Goldberg and Arkadi Vigalok*

Radicals generated during aldehyde oxidation to carboxylic acids can be efficiently trapped under environmentally friendly conditions, either in neat conditions or “on water”.



The aim of the conference is to highlight innovative concepts for the substitution of volatile organic solvents in solution phase synthesis. Emphasis will be laid on the development and application of alternative reaction media based on advanced fluids such as aqueous phases, ionic liquids, supercritical phases, green organic solvents, or soluble polymers, but includes also phase-separable reagents and related separation strategies in all areas of chemical synthesis.



October 10–13, 2010
Berchtesgaden/Germany

Green Solvents Conference

www.dechema.de/gsfs2010



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J.-D. Grunwaldt, KIT – Karlsruhe Institute of Technology/D
S.M. Howdle, University of Nottingham/UK
T. Ikariya, Tokyo Institute of Technology/J
P.G. Jessop, Queen's University, Kingston Ontario/CDN
U. Kragl, University of Rostock/D
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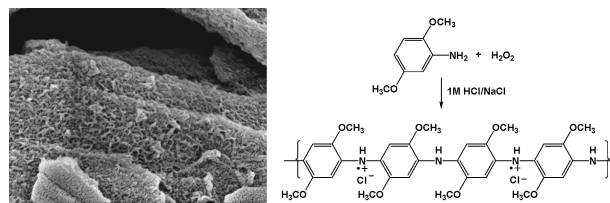


COMMUNICATIONS

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Green chemistry synthesis of nanostructured poly(2,5-dimethoxyaniline)

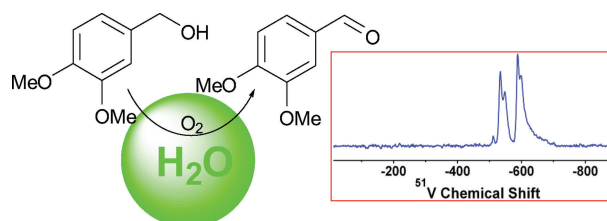
Sujit Jain, Sumedh P. Surwade, Srikanth Rao Agnihotra, Vineet Dua, Pamela A. Eliason, Gregory J. Morose and Sanjeev K. Manohar*

Bulk quantities of nanostructured poly(2,5-dimethoxyaniline) having high charge storage capacity are synthesized in one step using H_2O_2 as an environmentally friendly oxidant.

590

Synergistic effect of vanadium–phosphorus promoted oxidation of benzylic alcohols with molecular oxygen in water

Zhongtian Du, Jiping Ma, Hong Ma, Jin Gao and Jie Xu*

The synergistic effect of vanadium–phosphorus in aqueous solution is crucial to ensure high efficiency for $VOPO_4$ /TEMPO-catalyzed oxidation of benzylic alcohols with molecular oxygen in water.How can vanadium work for catalytic alcohol oxidation in H_2O ?

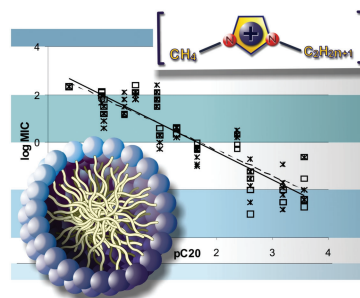
PAPERS

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Antimicrobial and surface activity of 1-alkyl-3-methylimidazolium derivatives

Justyna Łuczak, Christian Jungnickel,* Izabela Łącka, Stefan Stolte and Jan Hupka

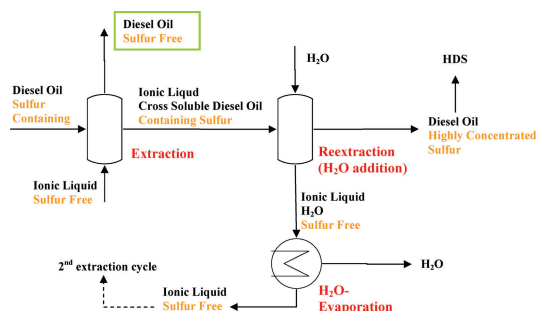
By analyzing 30 ionic liquids with 13 bacterial and fungal strains, we determined an antimicrobial mode of action dependant on the surface activity.



602

Desulfurization of diesel oil by selective oxidation and extraction of sulfur compounds by ionic liquids—a contribution to a competitive process design

Andreas Seeberger* and Andreas Jess

Efficient extraction of oxidized sulfur compounds from diesel oil by ionic liquids is combined with the continuous IL regeneration by addition of H_2O and the calculation of the overall energy demand.



2nd ASIA PACIFIC CONFERENCE ON IONIC LIQUIDS AND GREEN PROCESSES

September 7th-10th, 2010



Plenary Presentations



Tom Welton



Robin D. Rogers



Douglas MacFarlane



Zhang Suojiang



Han Buxing



Deng Youquan

Invited Talks



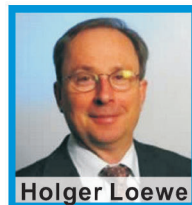
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Frank Endres



Axel Koenig



Holger Loewe



Giang VO THANH



Anil Kumar

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- Separation and Transport Processes
- Electrochemical and Energy Applications
- Sustainable and Environmental Technologies
- Process Engineering and Industrial Applications

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Abstract submission	April 30, 2010
Notification of abstract acceptance	May 31, 2010
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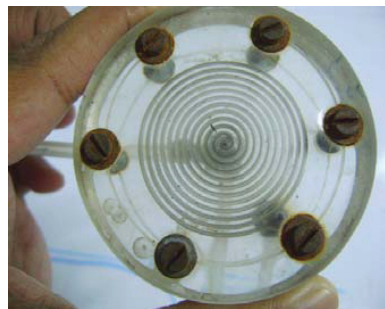
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Continuous flow synthesis of functionalized silver nanoparticles using bifunctional biosurfactants

D. V. Ravi Kumar, Manasi Kasture, A. A. Prabhune, C. V. Ramana, B. L. V. Prasad* and A. A. Kulkarni*

The continuous flow synthesis of silver nanoparticles using a biosurfactant and optimization of reaction conditions from the batch process was discussed.

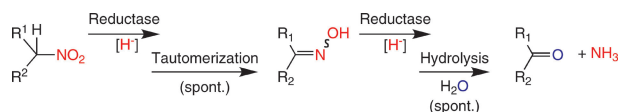


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The flavoprotein-catalyzed reduction of aliphatic nitro-compounds represents a biocatalytic equivalent to the Nef-reaction

Katharina Durchschein, Bianca Ferreira-da Silva, Silvia Wallner, Peter Macheroux, Wolfgang Kroutil, Silvia Maria Glueck and Kurt Faber*

The bioreduction of aliphatic *sec*-nitro compounds by flavoproteins from the old-yellow-enzyme family surprisingly furnished the corresponding carbonyl compounds instead of the expected amines and thus represents a biocatalytic equivalent to the Nef-reaction.

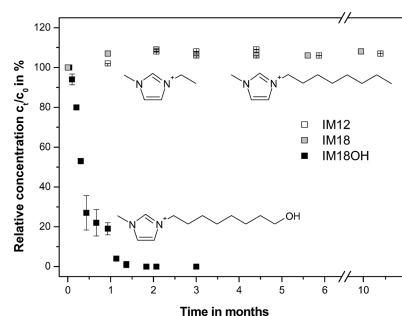


620

Anaerobic biodegradability of ionic liquid cations under denitrifying conditions

Jennifer Neumann, Olav Grundmann, Jorg Thöming, Michael Schulte and Stefan Stolte*

The primary anaerobic biodegradability of nine different imidazolium, pyridinium and dimethylaminopyridinium based IL cations has been examined for the first time. Only for the 1-(8-hydroxyoctyl)-3-methyl-imidazolium cation (IM18OH) a degradation could be observed and several metabolites were identified.

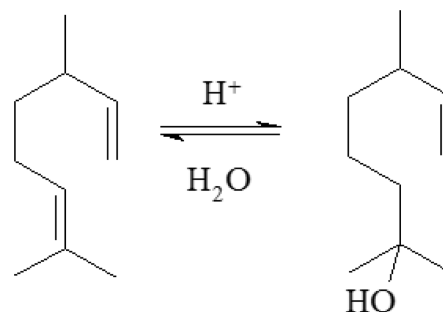


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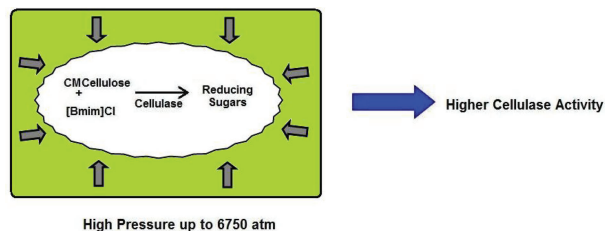
Selective hydration of dihydromyrcene in ionic liquids

Paul N. Davey, Martyn J. Earle,* Jennifer T. Hamill, Suhas P. Katdare, David W. Rooney and Kenneth R. Seddon

Acid catalysed direct hydration of dihydromyrcene to dihydromyrcenol (an important lily-of-the-valley fragrance) proceeds with 100% selectivity in certain ionic liquids.



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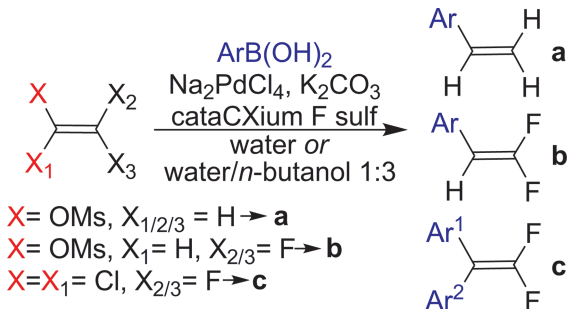


Effect of the ionic liquid [bmim]Cl and high pressure on the activity of cellulase

Ângelo C. Salvador, Mickael da C. Santos and Jorge A. Saraiva*

Cellulase shows 1.7-fold higher activity under high pressure (up to 600 MPa) in a buffer with 10% [bmim]Cl compared to atmospheric pressure, results that are interesting to improve enzymes' activity in ionic liquids using high pressure.

636

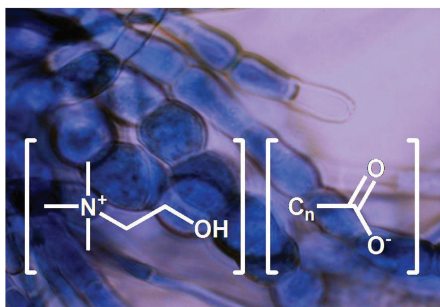


Cross coupling in water: Suzuki–Miyaura vinylation and difluorovinylation of arylboronic acids

Jan Pschierer, Natalie Peschek and Herbert Plenio*

An *in situ* formed Pd complex with a sulfonated fluorenylphosphine enables the vinylation and difluorovinylation of arylboronic acids *via* Suzuki–Miyaura coupling in water.

643

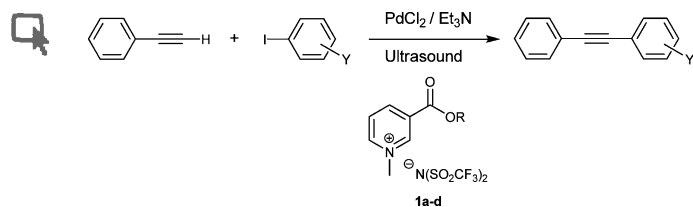


Novel biocompatible cholinium-based ionic liquids—toxicity and biodegradability

Marija Petkovic, Jamie L. Ferguson, H. Q. Nimal Gunaratne, Rui Ferreira, Maria C. Leitão, Kenneth R. Seddon, Luís Paulo N. Rebelo and Cristina Silva Pereira*

A toxicological assessment of the response of a series of novel cholinium alkanooates towards filamentous fungi (*Penicillium* sp.) highlights that conscious design of ionic liquids can lead to the development of truly biocompatible salts, carrying high biodegradability potential, without adversely affecting their outstanding solvent quality.

650



Sonogashira coupling reactions in biodegradable ionic liquids derived from nicotinic acid

Jitendra R. Harjani, Theodore J. Abraham, Alwyn T. Gomez, M. Teresa Garcia, Robert D. Singer* and Peter J. Scammells*

A series of biodegradable pyridinium ionic liquids have been evaluated as reaction media for copper- and phosphine-free Sonogashira coupling reactions. The stability of these ionic liquids toward basic conditions was analysed in order to further probe their utility for transition metal catalysed reactions which require the presence of a base.

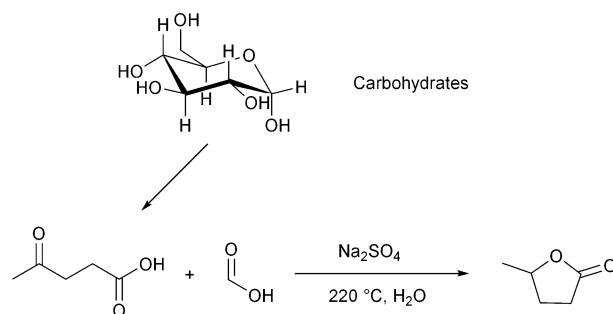
PAPERS

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Transfer hydrogenation of levulinic acid under hydrothermal conditions catalyzed by sulfate as a temperature-switchable base

Daniel Kopetzki* and Markus Antonietti

The transfer hydrogenation of biomass-derived levulinic acid with formic acid towards the biofuel molecule γ -valerolactone is performed in a simple hydrothermal reaction. Here, Na_2SO_4 serves as a temperature-switchable base to promote the reaction without additional catalysts.

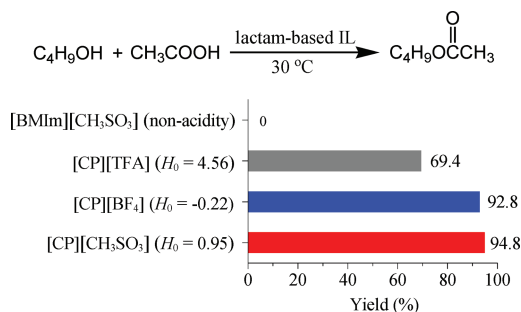


661

Effects of acidity and immiscibility of lactam-based Brønsted-acidic ionic liquids on their catalytic performance for esterification

Hancheng Zhou, Jing Yang, Linmin Ye, Haiqiang Lin and Youzhu Yuan*

Under mild conditions, lactam-based Brønsted-acidic ionic liquids having a methanesulfonate anion (which have weaker acidity than those with a tetrafluoroborate anion) exhibited higher catalytic performance and excellent reusability for esterification, indicating a synergistic effect of the acidity of ILs and their immiscibility with esters.

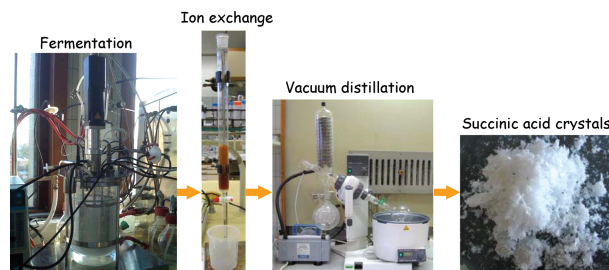


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Novel resin-based vacuum distillation-crystallisation method for recovery of succinic acid crystals from fermentation broths

Sze Ki Carol Lin,* Chenyu Du, Alexandra Cristina Blaga, Maria Camarut, Colin Webb, Christian V. Stevens and Wim Soetaert

This is the first publication to report the purity and yield of succinic acid crystals from actual fermentation broths using a novel resin-based vacuum distillation-crystallisation method.

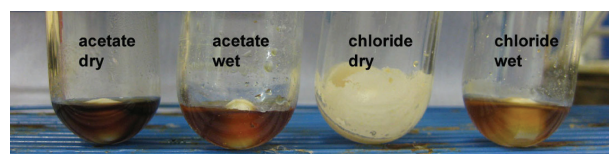


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The effect of the ionic liquid anion in the pretreatment of pine wood chips

Agnieszka Brandt, Jason P. Hallett, David J. Leak, Richard J. Murphy and Tom Welton*

The effect of the anion of ionic liquids on air-dried pine (*Pinus radiata*) has been investigated.



14th Annual Green Chemistry & Engineering Conference

Conference Topics

12 Principles
 Analytical Chemistry
 Biomaterials
 Biomimicry
 Coatings and Polymers
 Cosmetics and Personal Care
 Education
 Electronic Materials
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 Entrepreneurship
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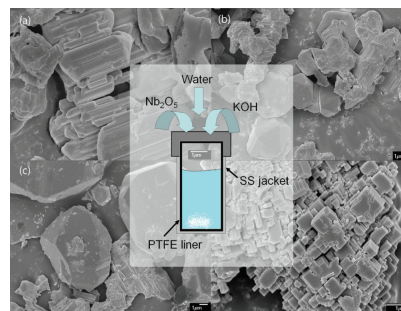
Co-sponsor of the "Green Jobs Act"

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Hydrothermal synthesis of sodium potassium niobate solid solutions at 200 °C

Albertus D. Handoko and Gregory K. L. Goh*

In this work we demonstrate the ability of hydrothermal synthesis in synthesizing sodium potassium niobate solid solution – previously only attainable at >1000 °C with solid-state synthesis – at 200 °C with controllable crystal size. This composition is important because it leads to the development of lead-free piezo/ferroelectric materials potentially replacing lead zirconium titanate (PZT).

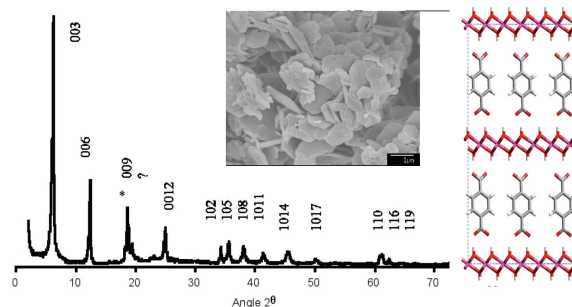


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Efficient synthesis of ordered organo-layered double hydroxides

H. Christopher Greenwell,* William Jones, Sarah L. Rugen-Hankey, Peter J. Holliman and Richard L. Thompson

An efficient method for the stoichiometric synthesis of layered double hydroxide catalysts is detailed which results in crystals with few surface defects and low size variation. X-Ray diffraction patterns show the material to be exceptionally highly ordered for organo-LDHs.

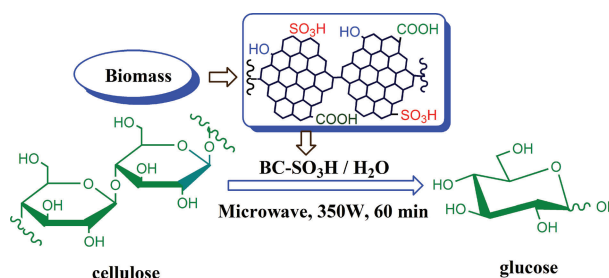


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Microwave-assisted hydrolysis of crystalline cellulose catalyzed by biomass char sulfonic acids

Youyu Wu, Zaihui Fu,* Dulin Yin,* Qiong Xu, Fenglan Liu, Chunli Lu and Liqiu Mao

An efficient and green process for the biomass char sulfonic acids (BC-SO₃H)-catalyzed hydrolysis of cellulose into reducing sugars in a small amount of water was achieved under microwave irradiation.

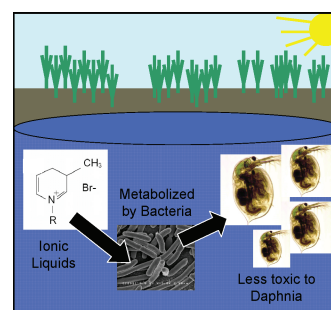


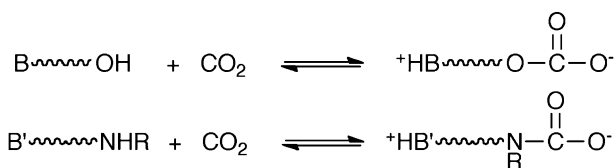
701

Microbial biodegradation and metabolite toxicity of three pyridinium-based cation ionic liquids

Kathryn M. Docherty,* Michelle V. Joyce, Konrad J. Kulacki and Charles F. Kulpa

Pyridinium-based ionic liquids are biodegraded *via* different pathways depending upon the length of the substituted alkyl chain. Biodegradation products are non-toxic to aquatic test organisms.





Reversible zwitterionic liquids, the reaction of alkanol guanidines, alkanol amidines, and diamines with CO₂

David J. Heldebrant,* Phillip K. Koech, M. Trisha C. Ang, Chen Liang, James E. Rainbolt, Clement R. Yonker and Philip G. Jessop

Alkanolamidines, alkanolguanidines and diamines each react with CO₂ to form reversible zwitterionic liquids. All three classes could be reverted to their non-ionic forms by thermally stripping CO₂ near 50 °C.

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