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GREEN CHEMISTRY

Reversible nonpolar-to-polar solvent

Imagine a smart solvent that can be switched reversibly from a liquid with one set of properties to another that has very different properties, upon command. Here we create such a system, in which a non-ionic liquid (an alcohol and an amine base) converts to an ionic liquid (a salt in liquid form) upon exposure to an atmosphere of carbon dioxide, and then reverts back to its non-ionic form when exposed to nitrogen or argon gas. Such switchable solvents should facilitate organic syntheses and separations by eliminating the need to remove and replace solvents after each reaction step.

Chemical production processes often involve multiple reaction and separation steps, and the type of solvent that is optimum for a particular step may be different from the one needed in the next step. The solvent is therefore usually removed after each step and a new solvent added in preparation for the next, significantly adding to the economic cost and environmental impact of the process. This cumbersome procedure would be unnecessary if a solvent's properties could be adjusted for the following step while still in the reaction vessel, enabling the same solvent to be used for several consecutive reaction or separation steps. Moderate changes in temperature and pressure are incapable of triggering significant changes in the properties of conventional solvents. In contrast, supercritical fluids[1] and CO_2/organic solvent mixtures[2] can be modified by pressure changes, but unfortunately only above 40 bar.

The reaction we describe reversibly changes the nature and properties of a solvent but occurs under very mild conditions. We reasoned that switching a normal non-ionic liquid to an ionic liquid should induce a change in its properties: ionic liquids are often viscous and always polar, whereas non-ionic solvents are typically non-viscous and vary widely in polarity. We chose CO_2 as the 'switch' to elicit this change because it is a benign agent and easily removed. (For methods, see supplementary information.)

We found that exposure of a 1:1 mixture of the two non-ionic liquids, namely DBU (1,8-diazabicyclo-[5.4.0]-undec-7-ene) and 1-hexanol, to gaseous CO_2 at one atmosphere and at room temperature causes conversion of the liquid mixture to an ionic liquid (Fig. 1a, b). This is readily converted back into a non-ionic liquid by bubbling N_2 or argon through the liquid at room temperature or, for a more rapid reaction, at 50 °C. These changes are demonstrated by chemical shifts in key protons, as revealed by ^{1}H-NMR spectroscopy, and by solvatochromic measurement of the polarity of the solvent before and after exposure to CO_2 (see supplementary information).

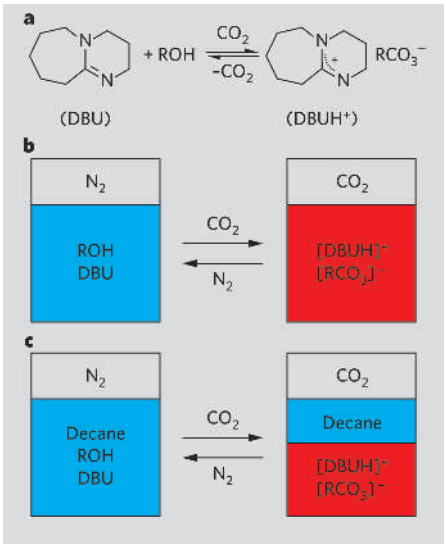

Figure 1 | The 'switching' of a switchable solvent. a, Protonation of DBU (1,8-diazabicyclo-[5.4.0]-undec-7-ene) in the presence of an alcohol and carbon dioxide is reversed when CO_2 is removed. **b,** Polarity switching in the reaction shown in **a**, in which CO_2 causes a nonpolar liquid (shown in blue) mixture of hexanol and DBU to change over one hour into a polar, ionic liquid (shown in red); nitrogen gas reverses the process by stripping out CO_2 from the reaction. **c,** The different polarity of each liquid under the two conditions is illustrated by the miscibility of decane with the hexanol/DBU mixture under nitrogen, before exposure to CO_2: however, decane separates out once the mixture becomes polar in the presence of CO_2. Again, N_2 reverses the process.

The reaction is exothermic and causes a marked increase in the viscosity of the liquid. The choice of alcohol is critical because the 1-hexylcarbonate salt (Fig. 1, right) is a viscous liquid at room temperature, whereas the bicarbonate[3,4] and methylcarbonate (ref. 5, and A. D. Main, G. E. Fryxell and J. Linehan, unpublished results) salts are solids and so are not candidates for smart solvents.

Our non-ionic liquid is as nonpolar as chloroform, according to measurements using Nile Red as solvatochromic dye (see supplementary information), whereas the liquid under CO_2 is as polar as dimethylformamide or propanoic acid. The polarity changes in this switchable solvent system are demonstrated by testing the solubility of decane, a nonpolar compound, in each liquid: it is miscible with the liquid under N_2 but not with that under CO_2 (Fig. 1c). We conclude that N_2 and CO_2 at 1 bar can be used as triggers of miscibility and immiscibility, respectively.

We have built solvent switchability into molecules that are small enough to be liquid at room temperature. Further examples of switchable solvents, preferably ones less Lewis-basic than DBU, should eventually enable their application in the 'green' production of high-value chemicals such as pharmaceuticals.

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Corrigendum

Dogs cloned from adult somatic cells

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This communication contains an error in the methods section of the supplementary information. In the description of the fusion protocol on page 3, line 2, electrical pulses were delivered for 15 microseconds, and not for 15 seconds as published.