How exotic synthesis path coupled with semi-automated processes can accelerate the discovery of new materials?

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As science has recently entered a new era in terms of knowledge quantity due to an unprecedented acceleration in scientific production mostly caused by the emergence of computational calculations and artificial intelligence, it is of the utmost importance to adapt research facilities to face these new challenges.

In recent years, several research groups have been developing their own facilities for the high throughput synthesis of materials for diverse applications [1]. This is also the case at the *CIC energiGUNE* research center, where a semi-automated laboratory has been developed for the synthesis and discovery of new materials applied to electrochemical energy storage, from active electrode materials to solid state electrolytes.

However, one of the issues with high throughput synthesis is the large number of samples that need to be characterized, moreover *operando and/or in situ* X-rays diffraction analyses are today not only common, but also essential to understand battery materials properties, which duplicates the number of required patterns refinements. These challenges motivated the development of the *FullProfAPP* software tool that facilitates full-profile phase searches and enables high-throughput Rietveld refinements, all via an intuitive graphical user interface [2].

The application of these tools can be extremely effective for the discovery of new materials, especially if they are coupled with exotic synthesis paths. The example of solvothermal synthesis using *Ionic Liquids* and *Deep Eutectic Solvents* has been chosen to illustrate the large scope of new possibilities using this method thanks to the dozens of millions different compositions of these solvents (Fig. 1) and the potentially new inorganic materials for electrochemical energy storage resulting from their use as solvents [3,4].

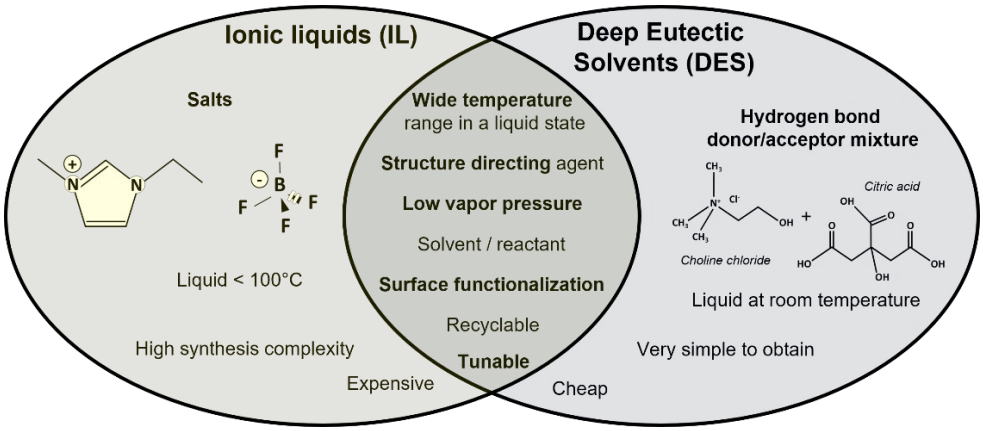

Figure 1. Illustration of the differences and complementarity of Ionic Liquids and Deep Eutectic Solvents

[1] McCalla, E. (2023) *ACS Eng. Au*, *3* (6), 391–402.

[2] Arcelus, O., Rodríguez-Carvajal, J., Katcho, N. A., Reynaud, M., Black, A. P., Chatzogiannakis, D., Carlos Fronterab, Jon Serrano-Sevillanoa, Maha Ismailae, Javier Carrascoaf, Francois Fauthg, M. Rosa Palacinb & Casas-Cabanas, M. (2024). *Applied Crystallography*, *57*(5).

[3] Minart, G., Duttine, M., Iadecola, A., Salvetat, J. P., Weill, F., Buffiere, S., Wernert R., Olchowka J. & Croguennec, L. (2024). *Chemistry of Materials*, *36*(20), 10186-10197.

[4] Minart, G., Fang, R., Labrugère-Sarroste, C., Weill, F., Buffiere, S., Cassaignon, S., Croguennec L. & Olchowka, J. (2025). *ACS Applied Materials & Interfaces*, *17*(10), 15301-15309.