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Mechanochemical synthesis of aromatics from biomass derived terpenes

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ABSTRACT

Over the past few decades, Green Chemistry has attracted attention of academia and industry due to safer activities towards humans and environment[1]. In this scenario, mechanochemistry has emerged as an enabling technology, matching high synergy with some principles of Green Chemistry, *e.g.* efficiency and sustainability [2]. Our research group has interest in the valorization of terpenes derived from biomass to synthesize phthalimides using RETSCH mixer mill MM 400. We commenced our study performing the Diels–Alder reaction between isoprene **(1)** and maleimides **(2)**. Afterwards, the adduct **(3)** undergoes aromatization in the presence of iodine and base, yielding phthalimides with yields around to 72% in solution and 89% in mechanochemistry conditions **(4)**.

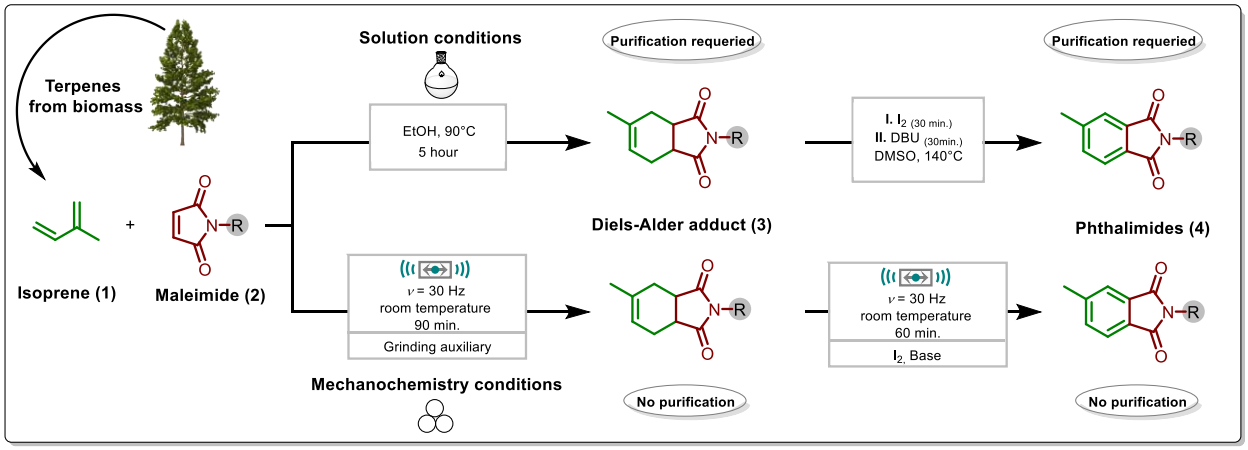

Figure 1. Conditions Diels-Alder and Aromatization Reaction

Compared to solution-based protocol, similar results are obtained in some advantages, like granting time saving, no use of bulk solvent use and no need of further purification according to Table 1.

Table 1. Conditions Aromatization Reaction

Solution conditions

Base	Time (min.)	Temperature (°C)	Solvent	Concentration (M)	Conversion (%)[1]	Yield (%)[1]
TMG	60	140	DMSO	0,1	100	48
DBU	60	140	DMSO	0,1	79	72

Mechanochemistry conditions

Base	Time (min.)	Temperature (°C)	Gringind Auxiliary	Jug* | Sphere*	Conversion (%)[1]	Yield (%)[1]
TMG	60	r.t	NaCl (2.5 mass eq.)	25 mL | 15mm	100	89
DBU	90	r.t	NaCl (2.5 mass eq.)	25 mL | 15mm	73	45

[1] Determined by NMR; *Stainless steel materials.

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