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Microwave Activation In Solvent Free Reaction

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Abstract:

For reasons of economy and pollution, solvent-free methods are of great interest to modernize classical procedures making them cleaner, safe and easy to perform. Reactions on solid mineral supports, reactions without any solvent/support or catalyst, and solid-liquid phase transfer catalysis can be thus employed with noticeable increases in reactivity and selectivity. A comprehensive review of these techniques is presented here. These methodologies can moreover be improved to take advantage of microwave activation as a beneficial alternative to conventional heating under safe and efficient conditions with large enhancements in yields and savings in time.

Keywords: Mineral solid supports, Phase transfer catalysis, Solid state reactions, Reactivity, Selectivity, Microwave activation.

Introductions:

Nowadays, one of the main duties assigned to the organic chemist is to organize research in such a way that it preserves the environment and to develop procedures that are both environmentally and economically acceptable. One significant objective is therefore to simplify and accommodate in a modern fashion the classical procedures with the purpose of limiting pollution effects to a minimum, combined with a reductionin energy and raw materials consumption.

Among the most promising ways to reach this goal, solvent-free techniques hold a strategic position as solvents are very often toxic, expensive, problematic to use and to remove. It is the main reason for the development of such modern technologies. These approaches can also enable experiments to be run without strong mineral acids (i. e. HCl, H2SO 4 for instance) that can in turn cause corrosion, safety, manipulation and pollution problems as waste. These acids can be substituted advantageously by solid, recyclable acids such as clays.

Microwave (MW) Activation in Solvent-Free Reactions

New strategies have recently been developed aimed at working without solvent. Furthermore, it is also possible to activate processes by physical means such as ultrasound, pressure or microwaves. Among these new non-conventional methods in organic synthesis, microwave irradiation takes a particular place as it induces interactions between materials and waves of an electromagnetic nature assimilated to dielectric heating. This original procedure involves heating the materials which then become reactive in situations where traditional treatments failed to give any reaction at all.

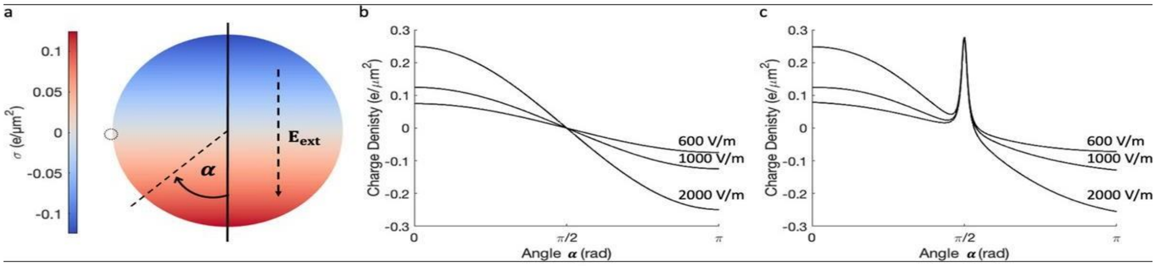
Generalities

Microwaves range from 1 cm (about 0.39 in) to 1 m in wavelength in the electromagnetic spectrum and are situated between the infrared and radio frequencies. The frequency band allowed by worldwide legislation, be it for industrial, scientific, medicinal or domestic purposes, corresponds to *u* = 2450 MHz (i.e. *l* = 12.2 cm (about 4.8 in) under vacuum*)*.The quantic energy involved can be evaluated to 0.3 cal/mol according to Planck's law E = h × c/l. This energy is far too low to induce any excitation of molecules or to provoke any reaction. The primary wave-material interactions are electromagnetic in nature, with a wavelength close to a few centimeters and a very significant depth of penetration into materials.

Microwave Heating

Heating of products submitted to microwave exposure can only result from material-wave interactions. It is brought about by the transformation into heat of a part of the energy contained in the electromagnetic wave. Polar molecules have the property that they can be orientated along an electric field (dipolar polarization phenomena). Dipole orientation is random in the absence of this phenomenon, and molecules are only subject to Brownian motion. All the dipoles are arranged in a straight line in the direction of a constant electric current. If submitted to an alternating current, the electric field is inversed at each

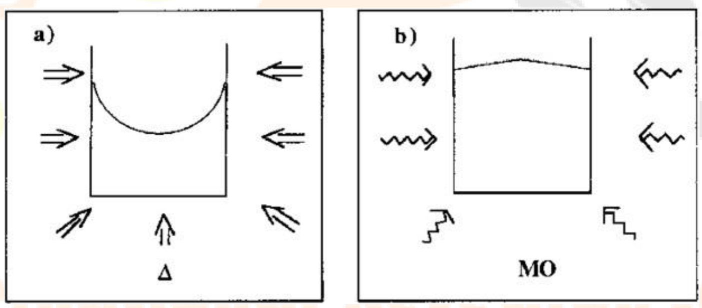
alternance with a subsequent tendency for dipoles to move together to follow the field. Such a feature produces churning and friction of molecules which dissipates as internal homogeneous heating.



Influence of electric field on a dielectric product

Advantages of Microwave Exposure

From the interactions between materials and electromagnetic waves heat is produced according to an original process characterized by a heating taking place in the core of the materials without superficial overheating, with a subsequent very homogeneous temperature. The profiles of gradients in temperature are inverted when going from classical heating (D) to microwave (MW) irradiation.

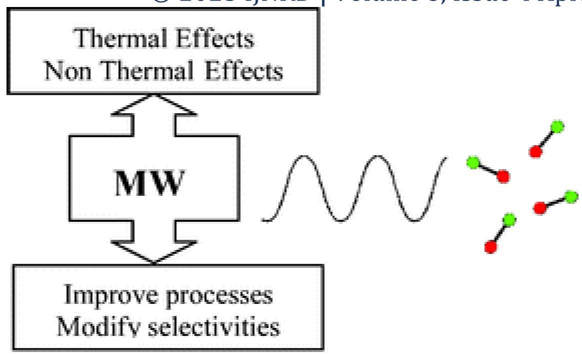


Gradient in temperature in solid submitted to (a) traditional heating by conduction (b) microwave exposure

Specific Effects of Microwaves (Purely Non-thermal)

Microwaves can be used to promote many chemical syntheses [105]. The mate-rials-wave interactions produce heating of the reaction medium by polar molecules (solvents, reagents or complexes, solid supports). To these purely thermal effects can be added specific effects due to MW radiation. To determine these effects a strict comparison is required between MW and conventional heating (*D*), all other conditions being identical (time, temperature, pressure, same profile of elevation in temperature). If the results obtained are different, the origin of these specific effects could be due to:

– a better homogeneity and speed of heating,
– the intervention of hot spots with high localized microscopic temperatures,
– variations in activation parameters $DG^{\neq} = DH^{\neq} - T \times DS^{\neq}$



Benefits

– very rapid reactions, frequently a few minutes, brought about by high and homogeneous temperatures and combined with pressure effects (if conduct-ed in closed vessels),
– higher degree of purity achieved due to short residence time at high temperatures, no local overheating, minor decomposition and minor occurrence of secondary reactions, and
– yields are often better, obtained within shorter times and with purer products.

Limitations:

The boiling points of solvents are reached rapidly, often posing safety problems (e.g. explosions). To solve these problems, the operation must be carried out in closed vessels (generally made of Teflon, a material transparent to MW and resistant up to 250°C and 80 psi) and using only small amounts of products (roughly 1/10 of the total volume). This of course constitutes a serious limitation (e.g. reduction in MW efficiency as the penetration depthis far below *l*, scaling up, etc.).

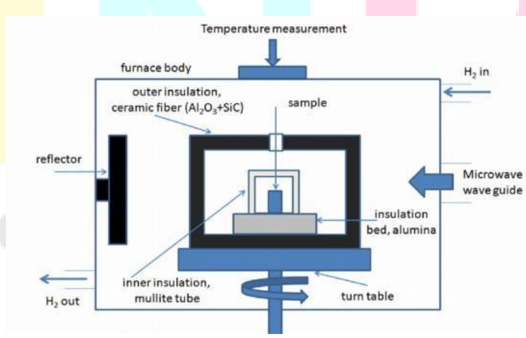
Another main limitation is the absence of measurement and control of temperature. However, these limitations can be overcome by the following two approaches:

1. to use solvent-free techniques,
2. to operate a monomodal reactor with permanent control of temperature.

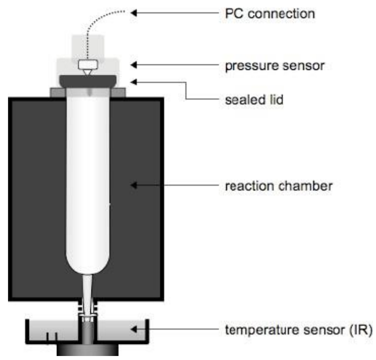
Equipment's:

Two types of reactors can be used in the laboratory:

– multimode systems

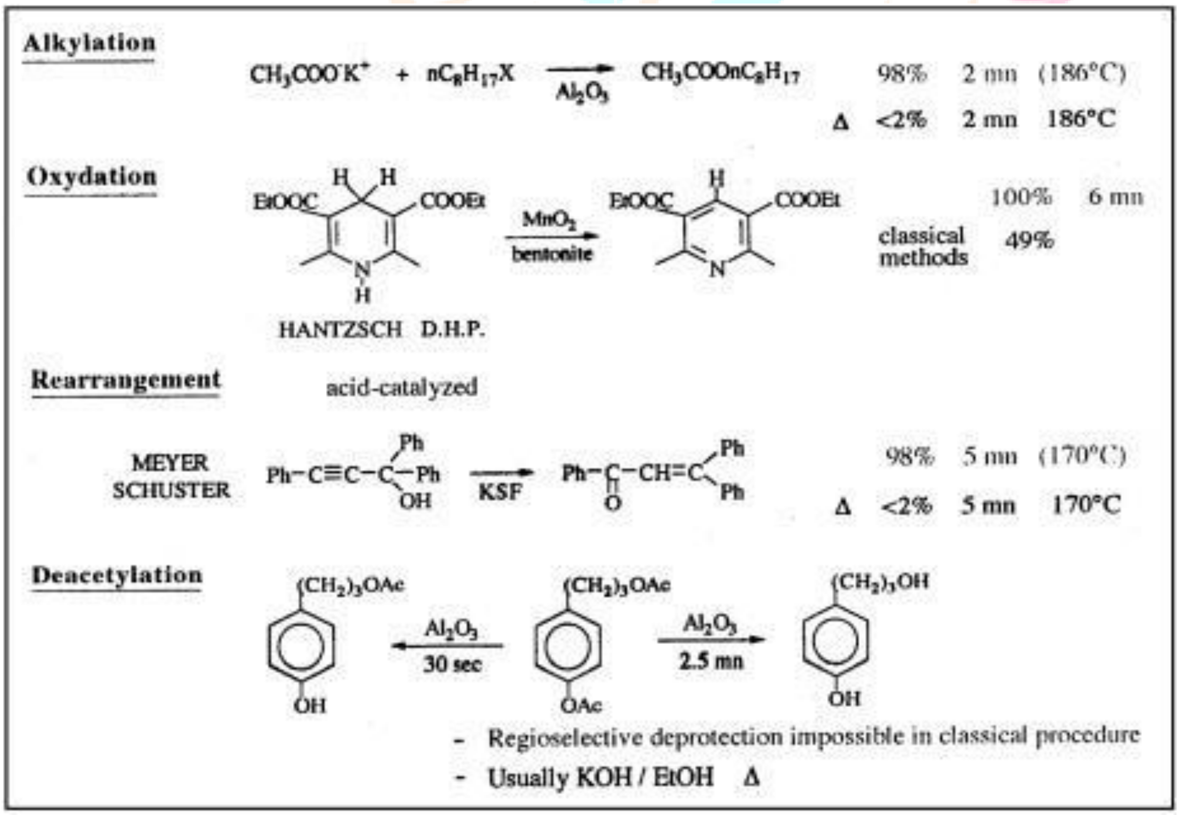


– monomode reactors



Solvent-free Organic Synthesis Under Microwaves:

It has been shown that solvent-free conditions are especially adaptable to micro-wave activation as reactions can be run safely under atmospheric pressure in the presence of significant amounts of products.



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