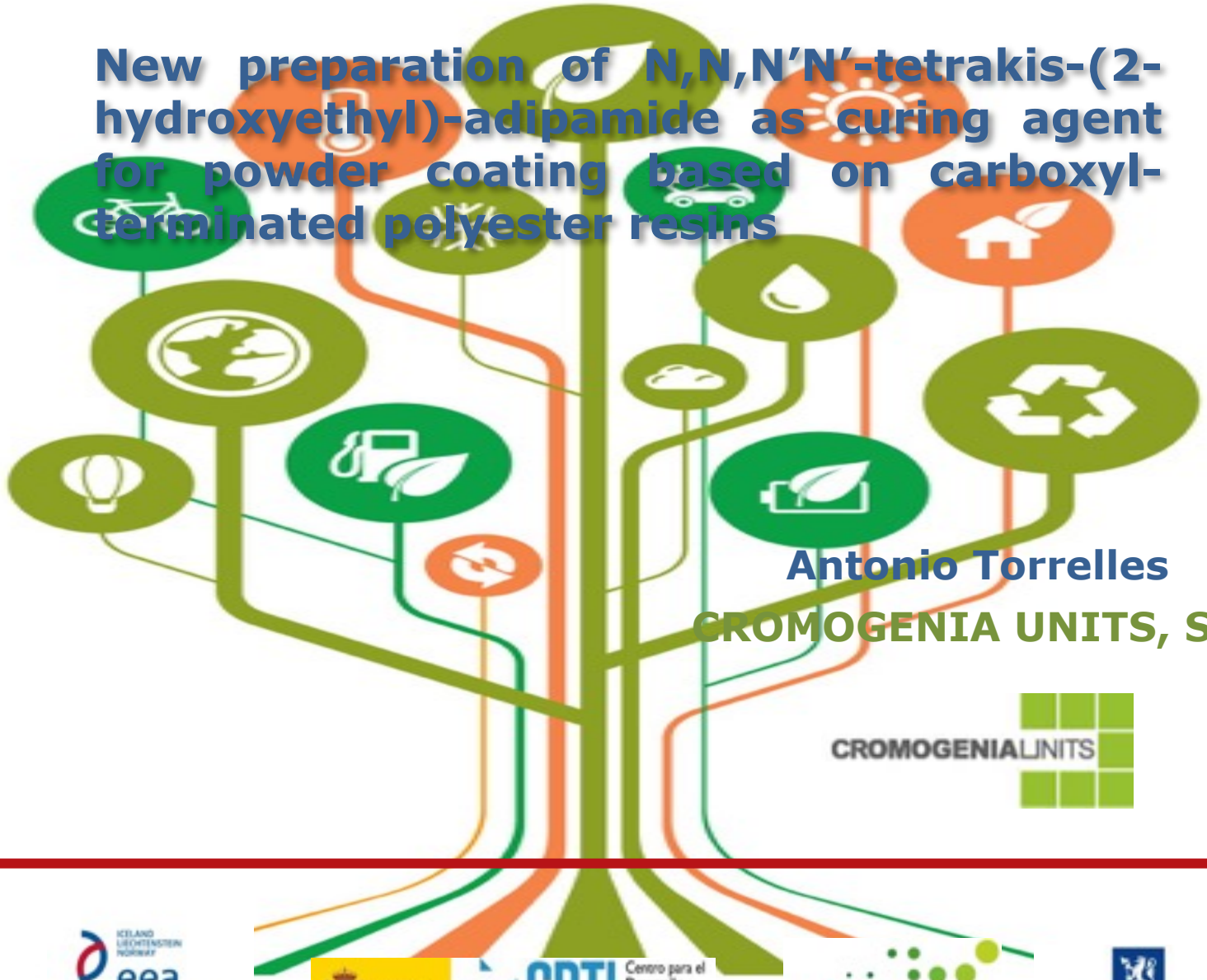
New preparation of N,N,N'N'-tetrakis-(2-hydroxyethyl)-adipamide as curing agent for powder coating based on carboxyl-terminated polyester resins



Antonio Torrelles

CROMOGENIA UNITS, SA

CROMOGENIAUNITS

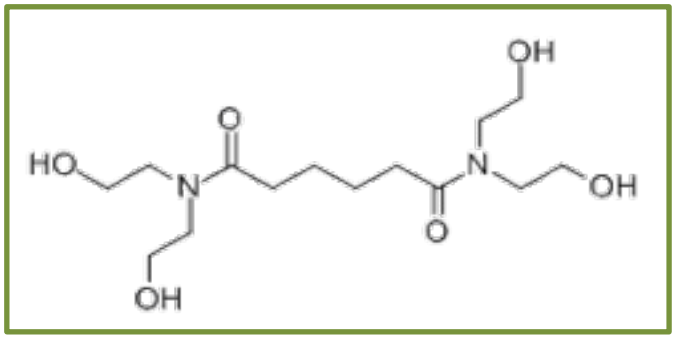
eea grants

CDTI Centro para el Desarrollo Tecnológico Industrial

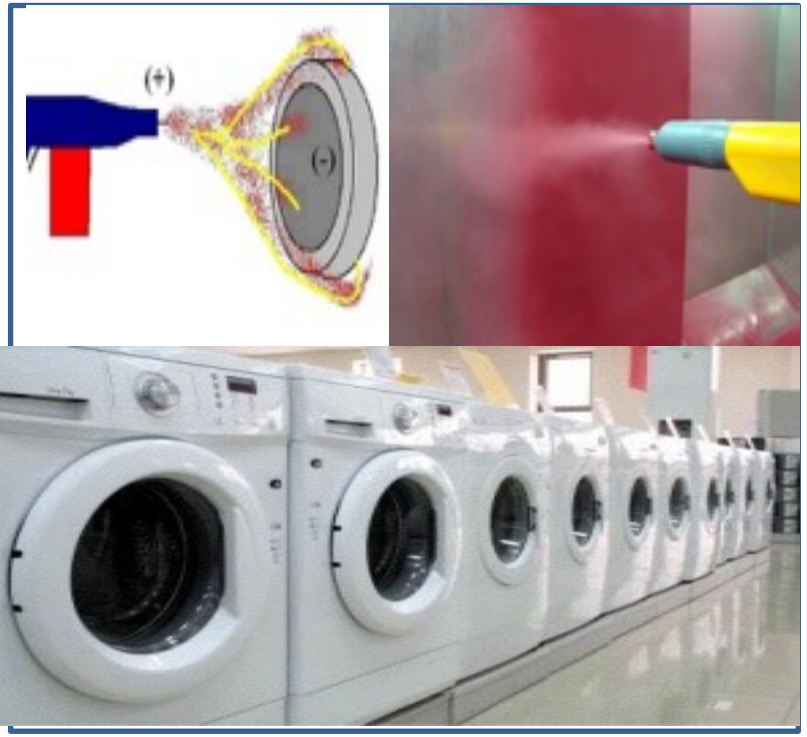
INNOVATION NORWAY

NORWEGIAN EMBASSY

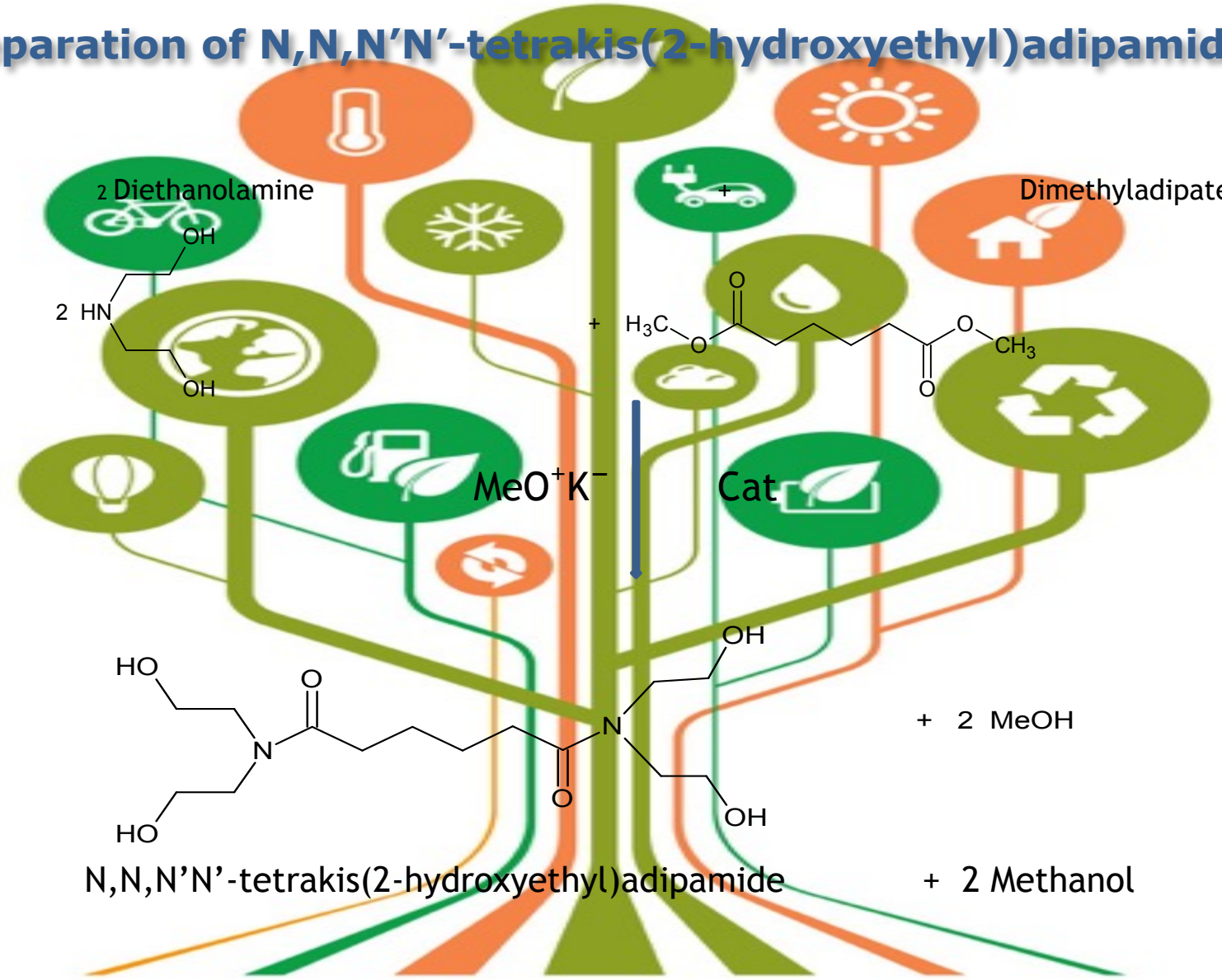
ROMID 320 is a 2-hydroxyalkyladipamide (2-HAA) crosslinker for the formulation of exterior durable powder coatings. It can be used in combination with carboxyl-terminated polyester. Meanwhile, triglycidyl isocyanurate (TGIC) is a toxic raw material. 2-HAA, like ROMID 320, is not classified as dangerous according to Directive 67/548/EEC.



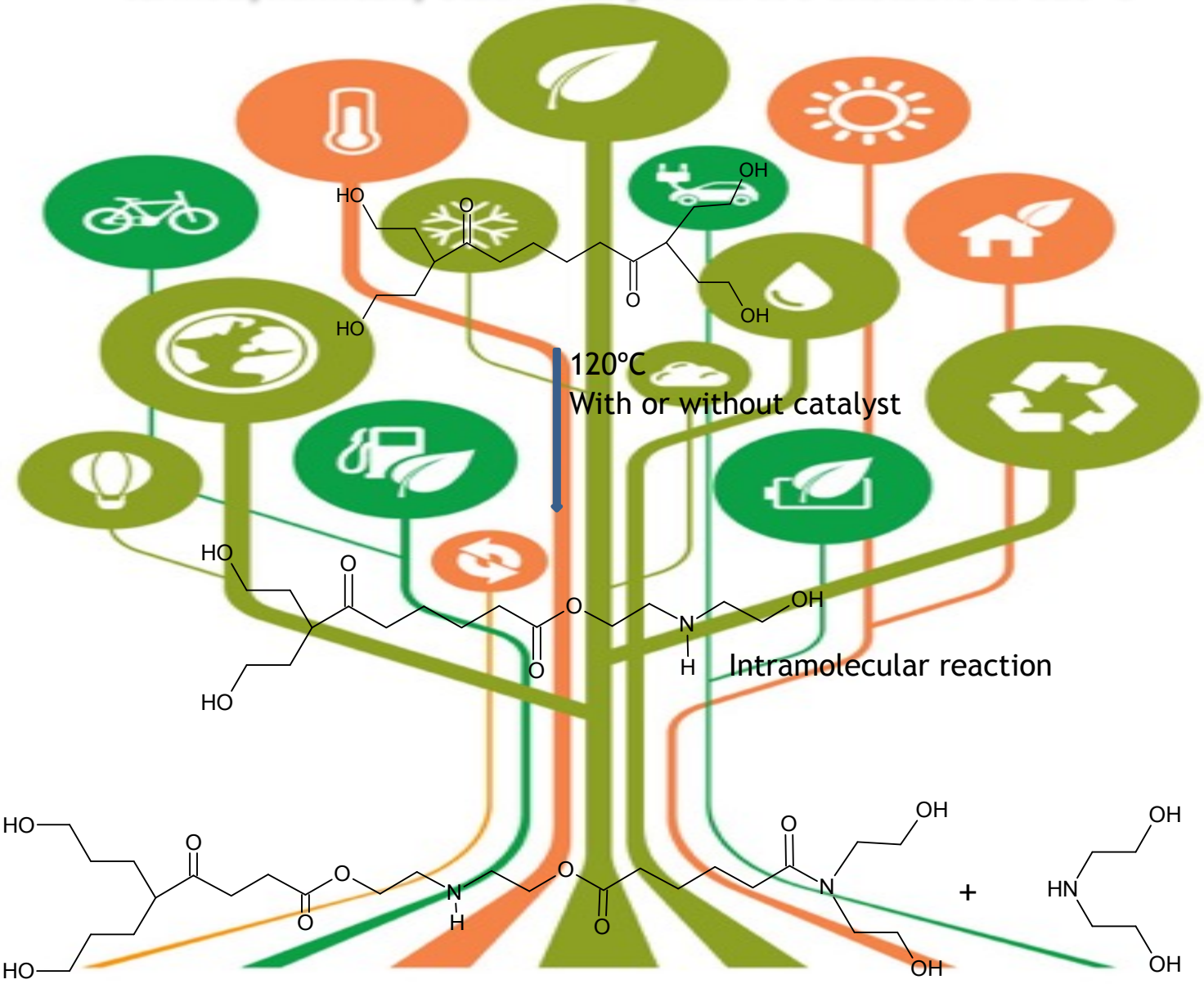
CAS Number: 6334-25-4
EC Number: 405-370-0
Molecular Formula: C14H28N2O6
Melting Point: 124/128ºC



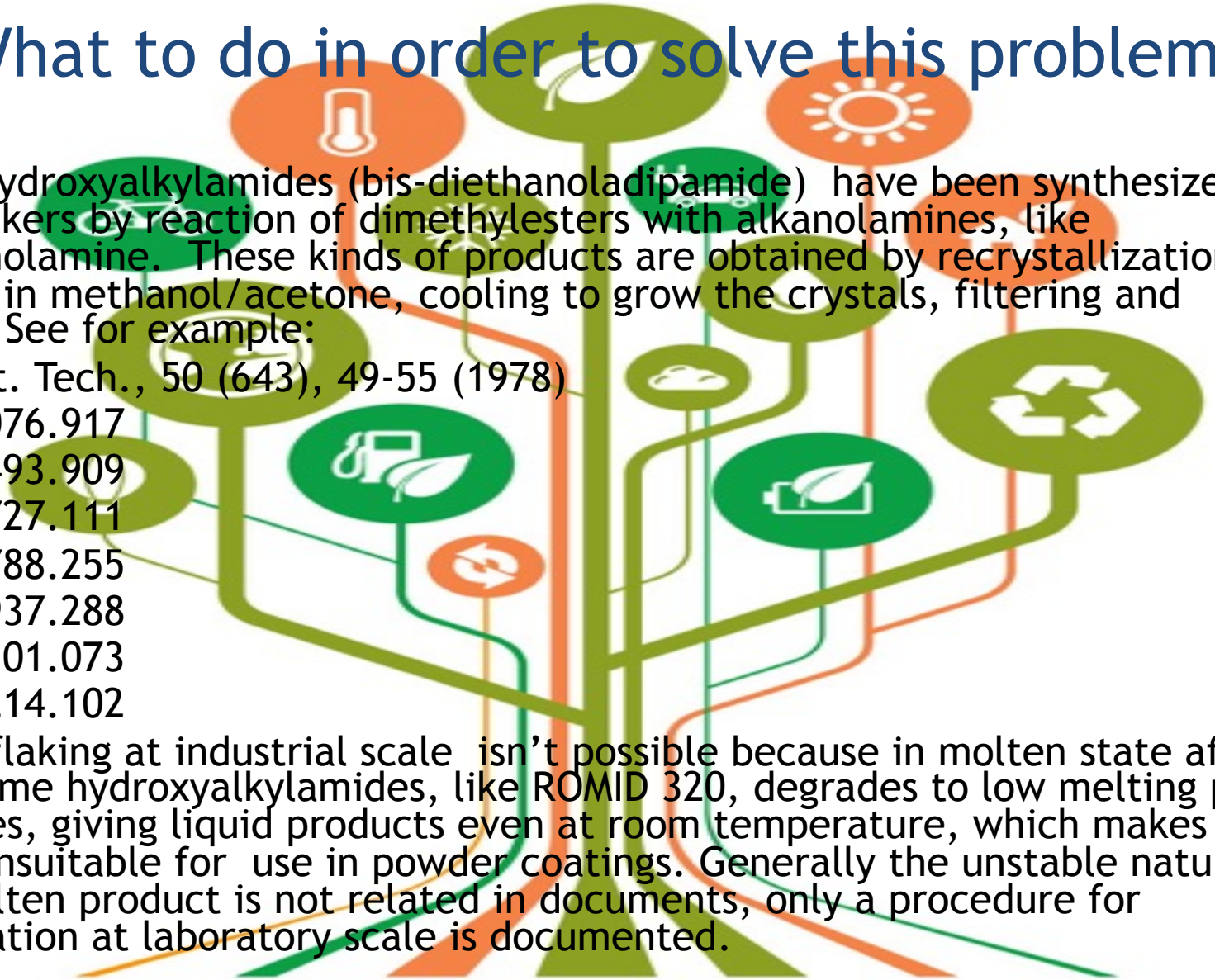
Preparation of N,N,N'N'-tetrakis(2-hydroxyethyl)adipamide



N,N,N'N'-tetrakis(2-hydroxyethyl)adipamide is not a termodynamically stable compound. It's unstable at 120ºC



What to do in order to solve this problem?

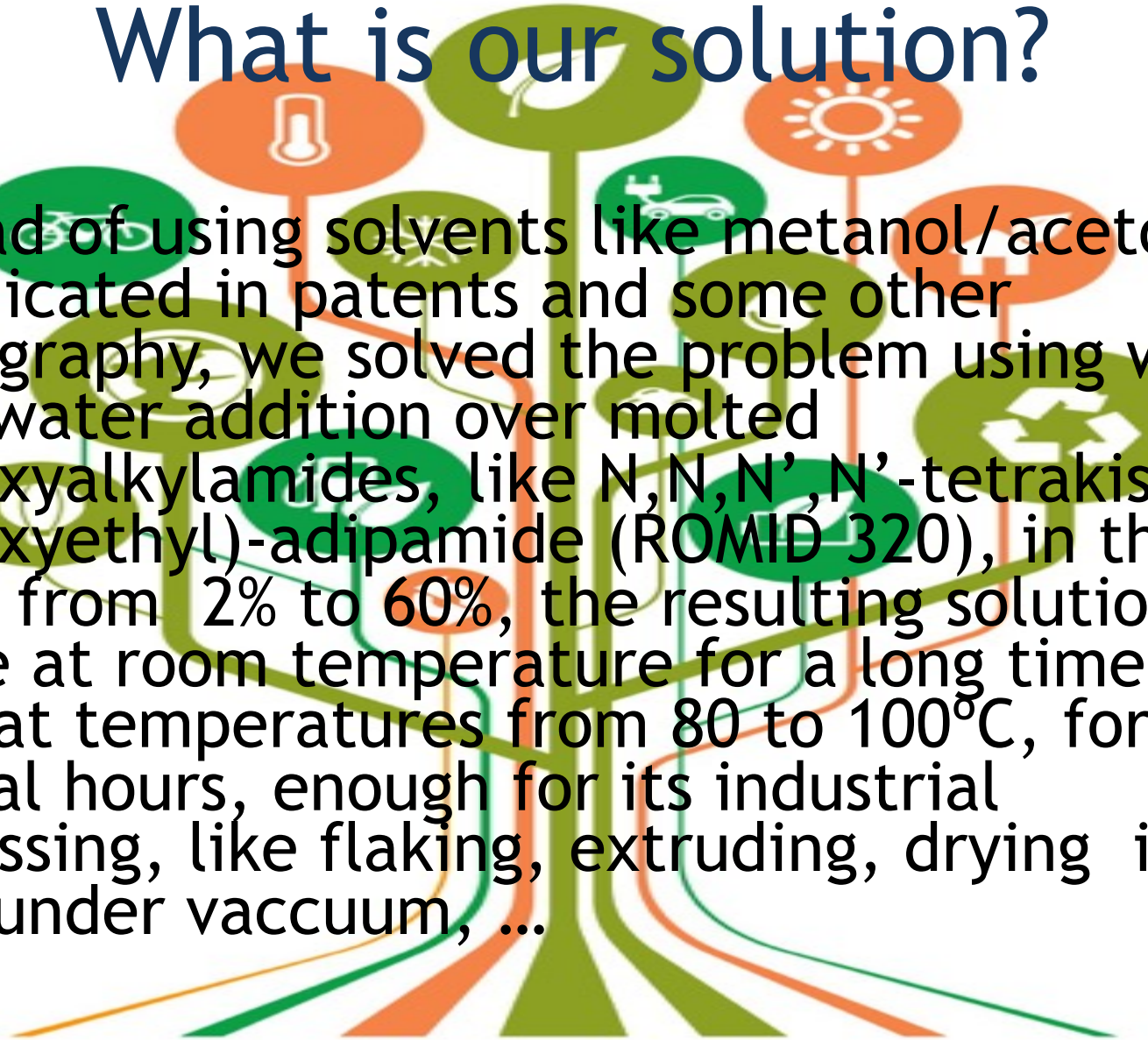


Some hydroxyalkylamides (bis-diethanoladipamide) have been synthesized as crosslinkers by reaction of dimethylesters with alkanolamines, like diethanolamine. These kinds of products are obtained by recrystallization, usually in methanol/acetone, cooling to grow the crystals, filtering and drying. See for example:

J. Coat. Tech., 50 (643), 49-55 (1978)
USP 4.076.917
USP 4.493.909
USP 4.727.111
USP 4.788.255
USP 4.937.288
USP 5.101.073
USP 5.214.102

Direct flaking at industrial scale isn't possible because in molten state after short time hydroxyalkylamides, like ROMID 320, degrades to low melting point mixtures, giving liquid products even at room temperature, which makes them unsuitable for use in powder coatings. Generally the unstable nature of the molten product is not related in documents, only a procedure for preparation at laboratory scale is documented.

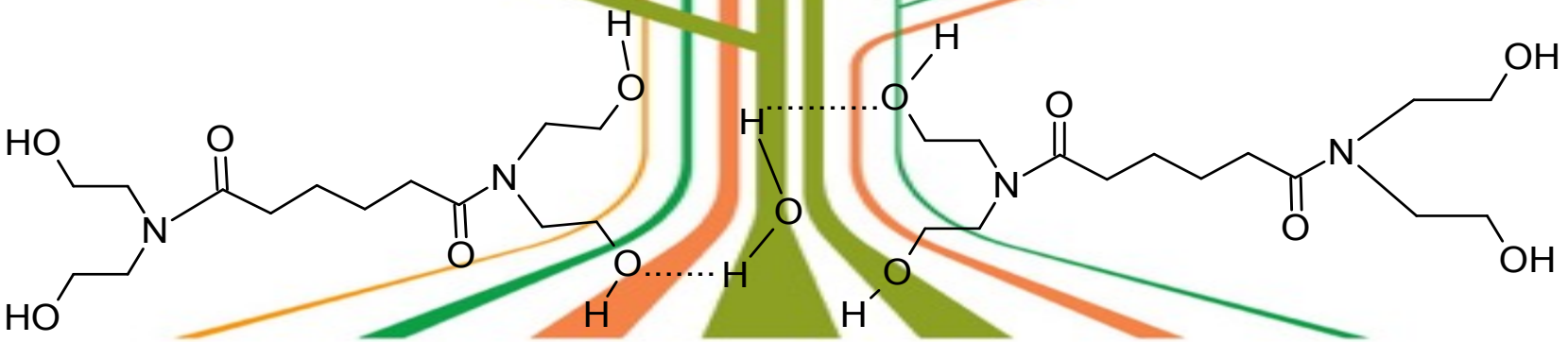
What is our solution?

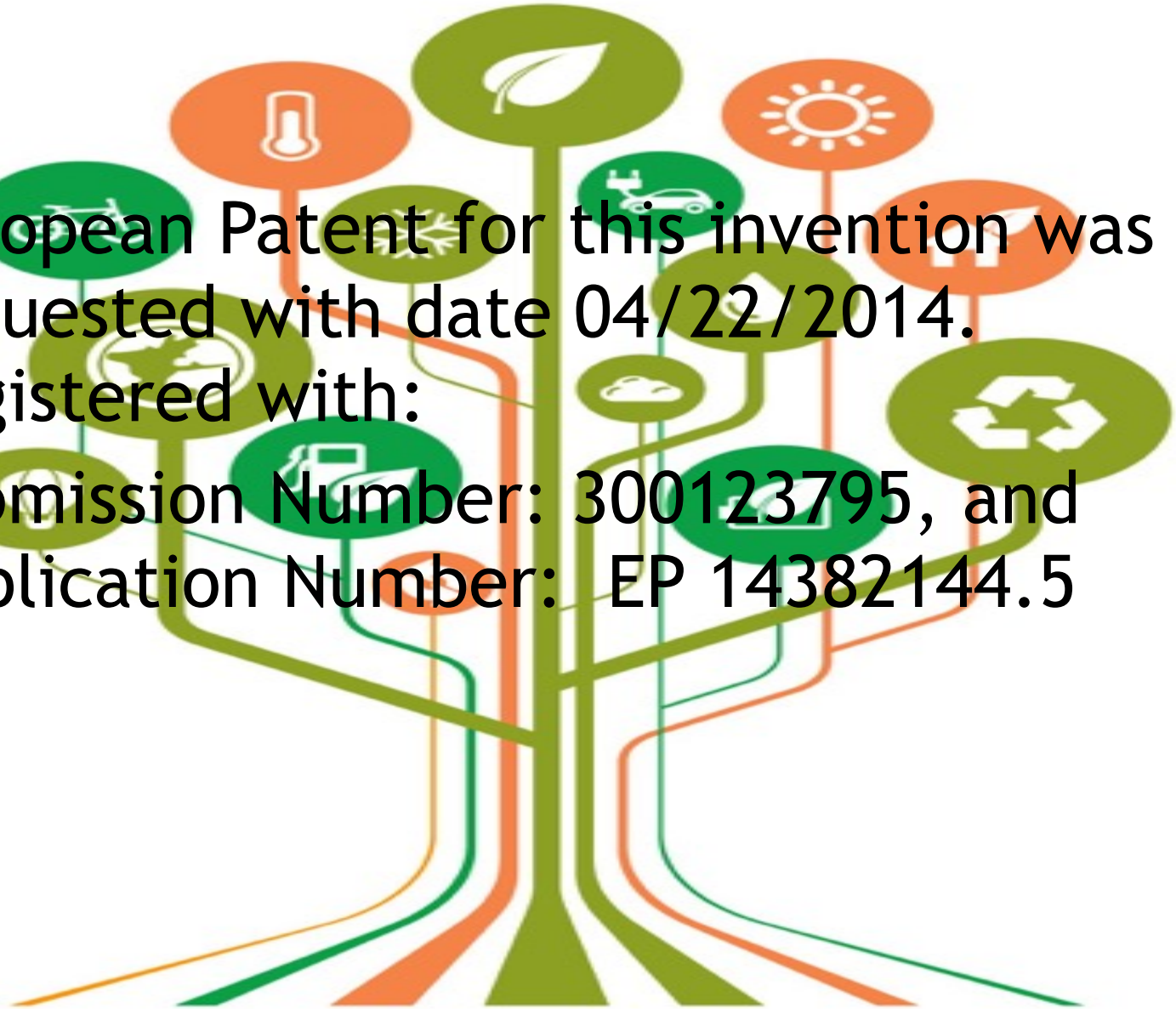


Instead of using solvents like metanol/acetone, as indicated in patents and some other bibliography, we solved the problem using water. With water addition over molted hydroxyalkylamides, like N,N,N',N'-tetrakis-(2-hydroxyethyl)-adipamide (ROMID 320), in the range from 2% to 60%, the resulting solution is stable at room temperature for a long time, and even at temperatures from 80 to 100ºC, for several hours, enough for its industrial processing, like flaking, extruding, drying in an oven under vaccuum, …

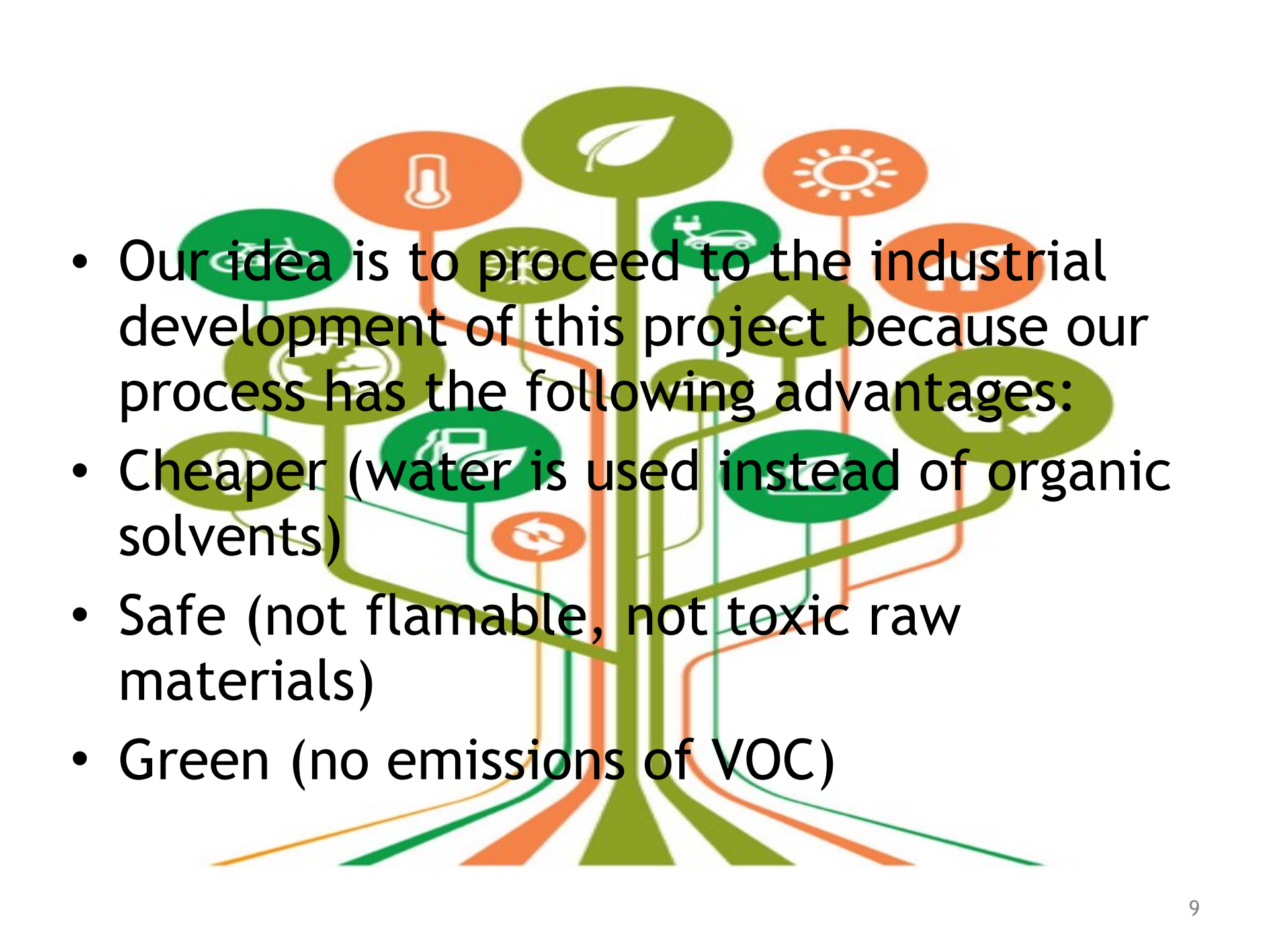
By addition of water to 2-hydroxyalkylamides, N,N,N’N’-tetrakis-(2-hydroxyalkyl)-adipamide is stabilized by hydrogen bonds between water and the amide hydroxyl groups.

Hydrogen Bonds in molar ratio 2:1





- European Patent for this invention was requested with date 04/22/2014. Registered with:
- Submission Number: 300123795, and Application Number: EP 14382144.5



- Our idea is to proceed to the industrial development of this project because our process has the following advantages:
- Cheaper (water is used instead of organic solvents)
- Safe (not flamable, not toxic raw materials)
- Green (no emissions of VOC)