New Journal of Chemistry

Supporting information

Talc-like hybrids: influence of the synthesis parameters on the polycondensation degree, crystallinity and thermal stability

Marion BRUNEAU[1,2,3], Jocelyne BRENDLE*[1,2], Simona BENNICI*[1,2], Lionel LIMOUSY[1,2], Sylvain PLUCHON[3]

[1]*Université de Haute-alsace, CNRS, IS2M UMR 7361, F-68100 Mulhouse, France*
[2]*Université de Strasbourg, France*
[3]*Agro Innovation International, 35400 Saint Malo, France*

* *Corresponding authors at: Université de Haute-Alsace, CNRS, IS2M UMR 7361, F-68100 Mulhouse, France ; Email:* jocelyne.brendle@uha.fr

I. XRD results

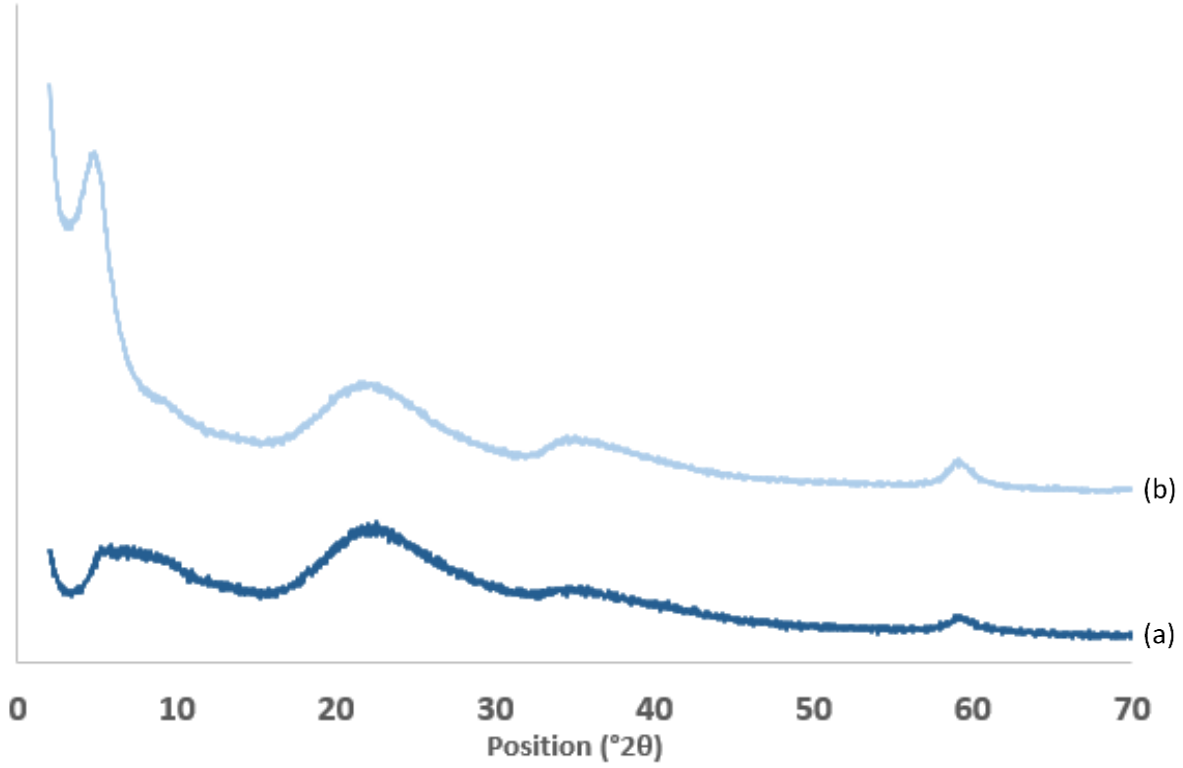

Figure A : XRD patterns of the talc-like hybrids synthetized in phenoxy-2-propanol (a) and absolute ethanol (b)

In figure A, the XRD pattern of the hybrids synthetized in phenoxy-2-propanol and absolute ethanol shows the five main reflections typical of talc-like hybrids. It can be noticed in the first diffractogramme (a) that the intensity of the (001) reflection is lower and the thickness is higher than in the second one (b), indication of a low degree of crystallinity for the hybrid synthetized in presence of phenoxy-2-propanol.

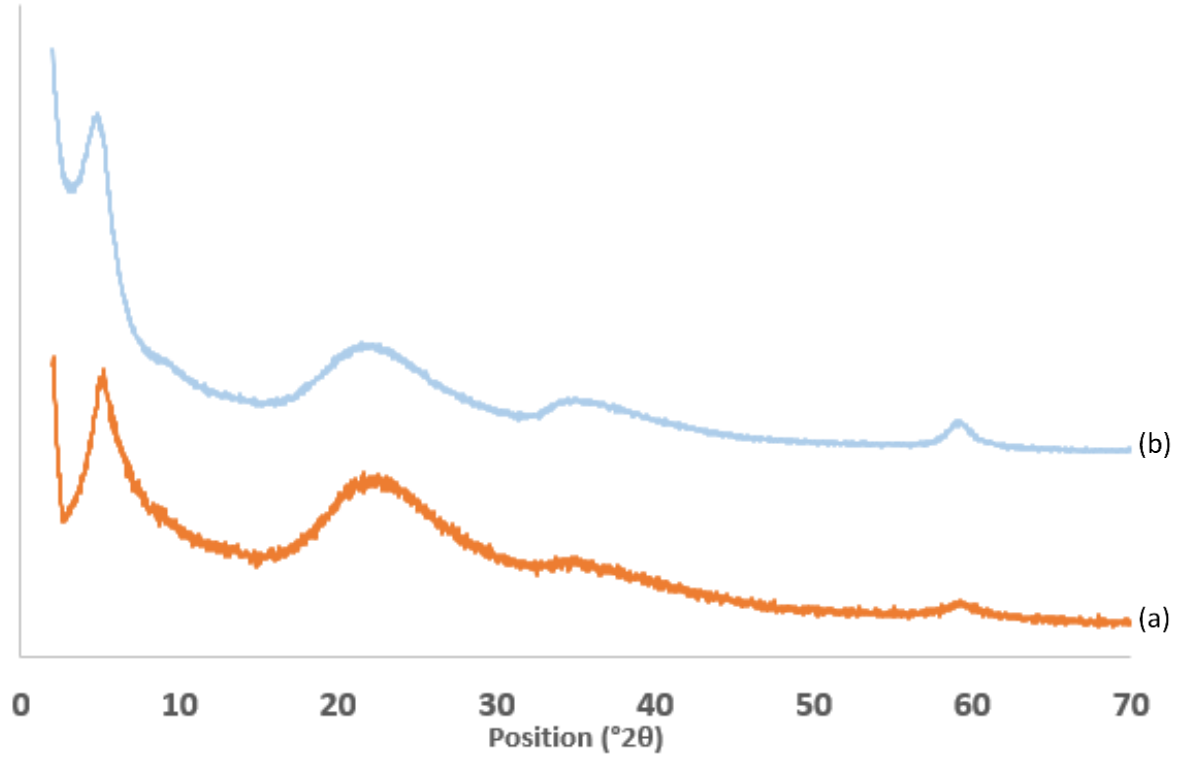

Figure B : XRD patterns of the talc-like hybrids synthetized during 1 day (a) and 5 days (b)

In figure B, the XRD pattern of the hybrid synthesized during 5 days presents a more intense (001) reflection that the pattern of the hybrid synthesized during one day that indicate a higher degree of crystallinity. This phenomenon can be explained by a longer hydrolysis-condensation process time which favours the formation of talc-like hybrids.

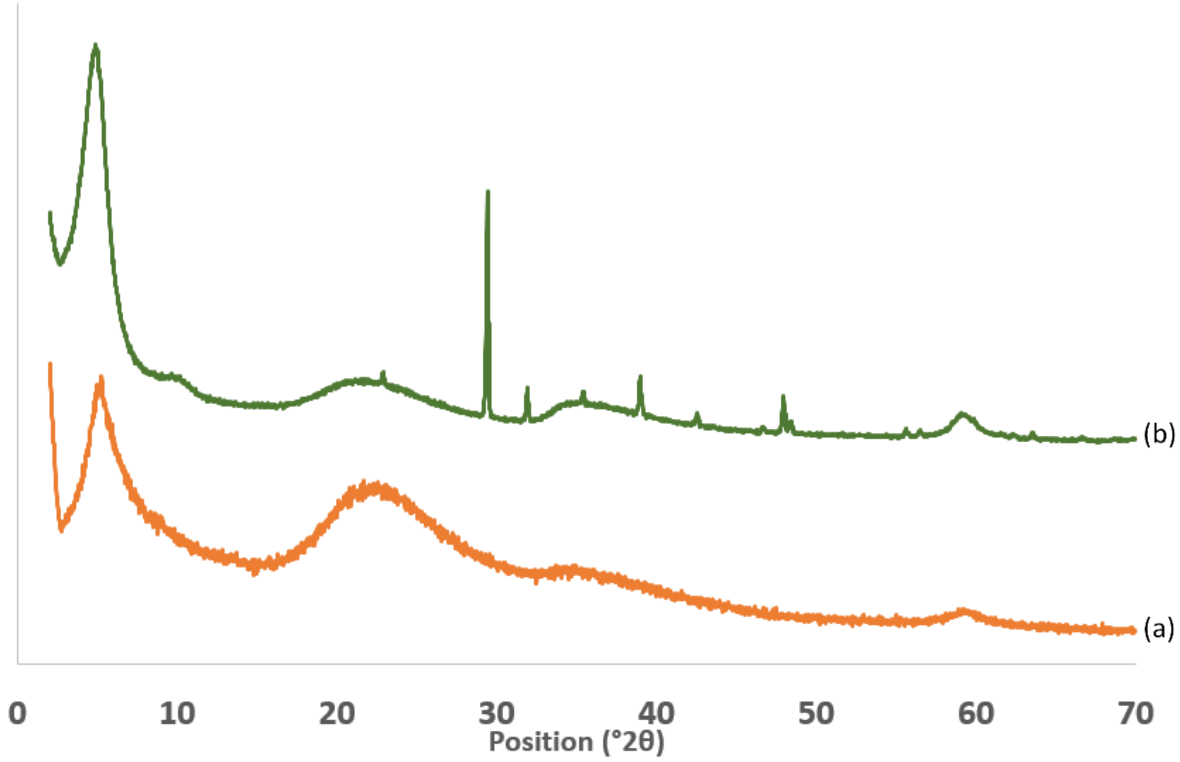

Figure C : XRD patterns of the talc-like hybrids synthetized in absence of NaOH (a) and in presence of NaOH (b)

In figure C, the XRD pattern of the hybrid synthesized in presence of sodium hydroxide presents a more intense (001) reflection than the XRD pattern of the hybrid synthesized in absence of NaOH and that indicate a higher degree of crystallinity for this hybrid compare to the other. This phenomenon can be explained by the enhanced condensation process due to the high pH of the synthesis medium. The narrow reflections that can be observed in the XRD pattern of the hybrid synthetized in presence of NaOH are attributed to sodium nitrate, a by-product of the reaction between magnesium nitrate and sodium hydroxide.

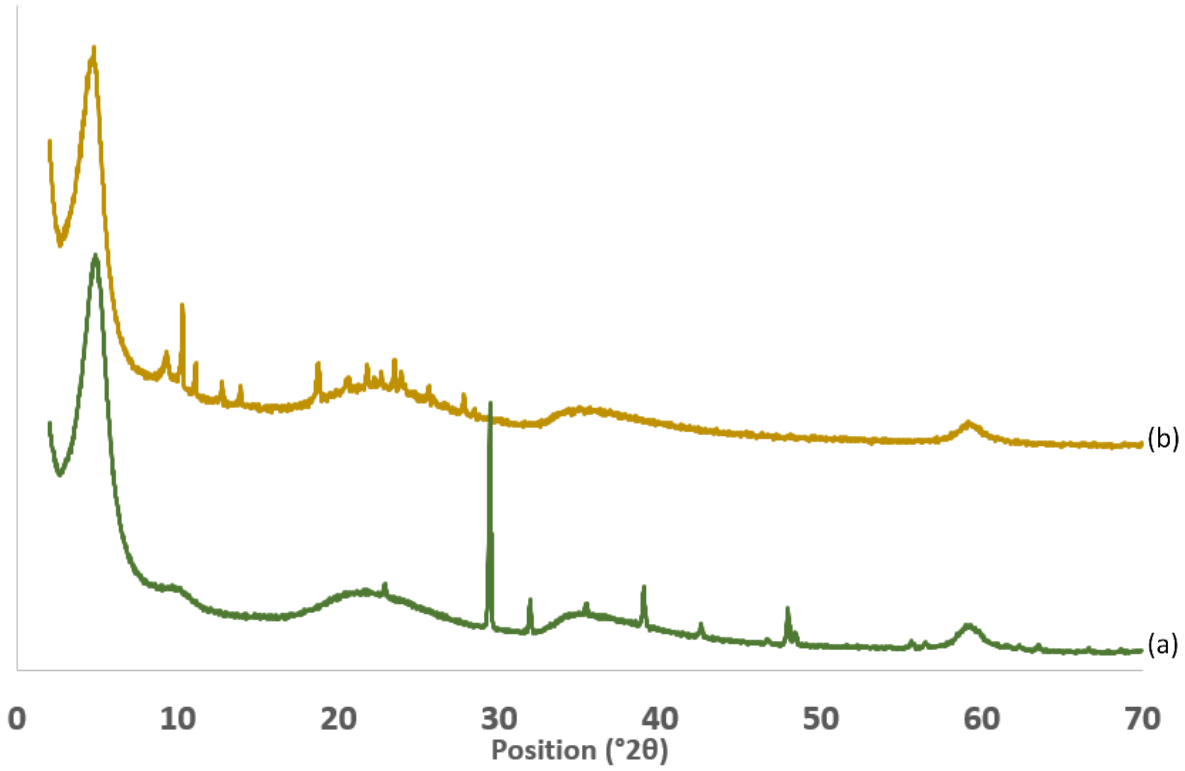

Figure D : XRD patterns of the talc-like hybrids synthetized in presence of NaOH (a) or TPAOH (b)

In figure D, the two XRD patterns are very similar indicating that the nature of the base used in the synthesis medium has no influence on the crystallinity of the hybrids. The narrow reflections that can be observed in the XRD pattern of the hybrid synthetized in presence of NaOH are attributed to sodium nitrate, a by-product of the reaction between magnesium nitrate and sodium hydroxide. The narrow peaks observed in the XRD pattern of the hybrid synthetized in presence of TPAOH are not identified, but can be reasonably attributed to an impurity, formed thanks to the addition of TPAOH.

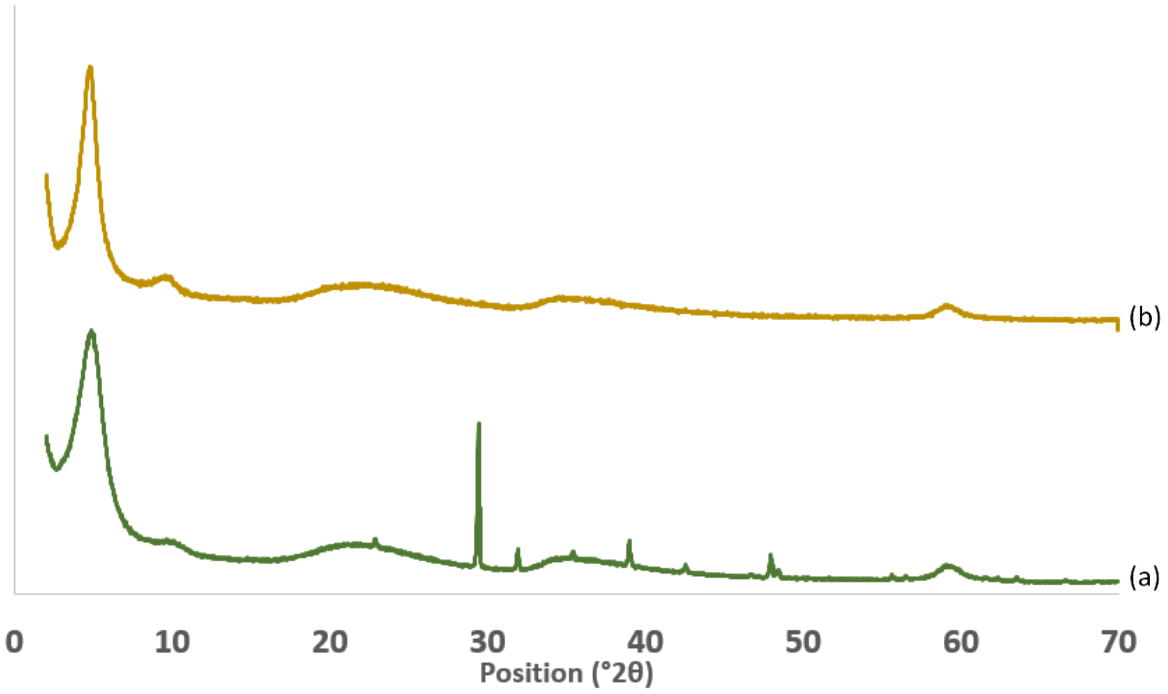

Figure E : XRD patterns of the talc-like hybrids synthetized in presence of NaOH (a) or water (b)

In figure E, the two XRD patterns are very similar indicating that the presence of sodium hydroxide or water has the same influence on the crystallinity of the hybrids. The narrow reflections that can be observed in the XRD pattern of the hybrid synthetized in presence of NaOH are attributed to sodium nitrate, a by-product produced during the reaction between magnesium nitrate and sodium hydroxide.

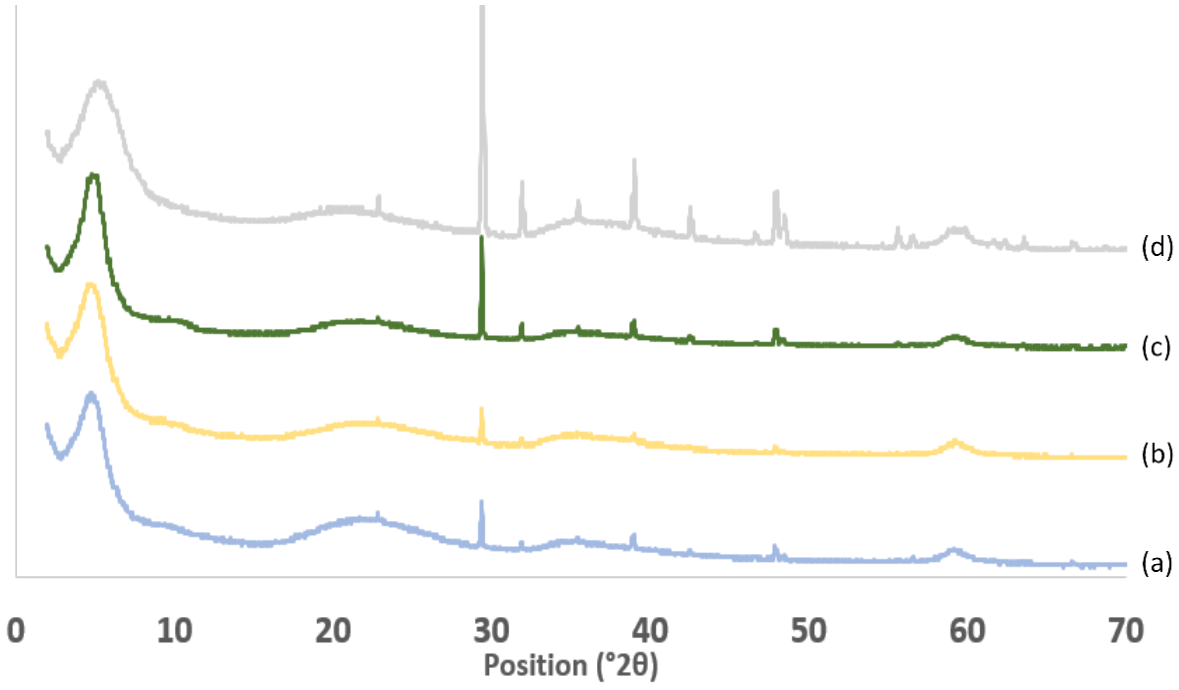

Figure F : XRD patterns of the talc-like hybrids synthetized at different pH : pH=9, 4 mL of NaOH 1 M (a); pH=9, 8 mL of NaOH 0.5 M (b), pH=10, 8 mL of NaOH 1 M (c) and pH=13, 8 mL of NaOH 2 M (d)

In figure F, the four XRD patterns are similar indicating that the pH does not have a strong influence in the crystallinity of the hybrids. Only the hybrid synthetized at pH=13 shows a small decrease of the crystallinity. The narrow reflections that can be observed in the XRD pattern of the hybrid synthetized

in presence of NaOH are attributed to sodium nitrate, a by-product of the reaction between magnesium nitrate and sodium hydroxide.

II. TGA results

Table A : Experimental ratio of organic species in the hybrids according to TGA analysis. The theoretical value is 49.0 %

Condition of synthesis (duration, solvent, others)	Organic moieties ratio (%)
Hybrid, 5 days, in phenoxy-2-propanol	75.5
Hybrid, 5 days, in ethanol	48.2
Hybrid, 1 day, in ethanol	47.8
Hybrid, 1 day, in ethanol, with 8 mL of NaOH 0.5 M (pH=9)	44.1
Hybrid, 1 day, in ethanol, with 4 mL of NaOH 1 M (pH=9)	44.9
Hybrid, 1 day, in ethanol, with 8 mL of NaOH 1 M (pH = 10)	45.0
Hybrid, 1 day, in ethanol, with 8 mL of TPAOH 1 M (pH=10)	48.9
Hybrid, 1 day, in ethanol, with 8 mL of NaOH 2 M (pH=13)	40.4
Hybrid, 1 day, in ethanol, with 4 mL of water	47.8
Hybrid, 1 day, in ethanol, with 8 mL of water	45.9
Hybrid, 1 day, in ethanol, with 12 mL of water	45.0

The hybrid synthetized in phenoxy-2-propanol present a high organic ratio because, even after washing, some solvent remains in the material. The hybrid synthetized in presence of TPAOH have also a high ratio. This can be explained by the impurity present in the material that can be observed by XRD. All the others have got ratios below the theoretical value due to the carbonaceous residue remaining in the sample after TGA.