



MORPHOLOGICAL PECULIARITIES OF ISOTACTIC POLYPROPYLENE NUCLEATED WITH ALKALINE EARTH METAL PIMELATES

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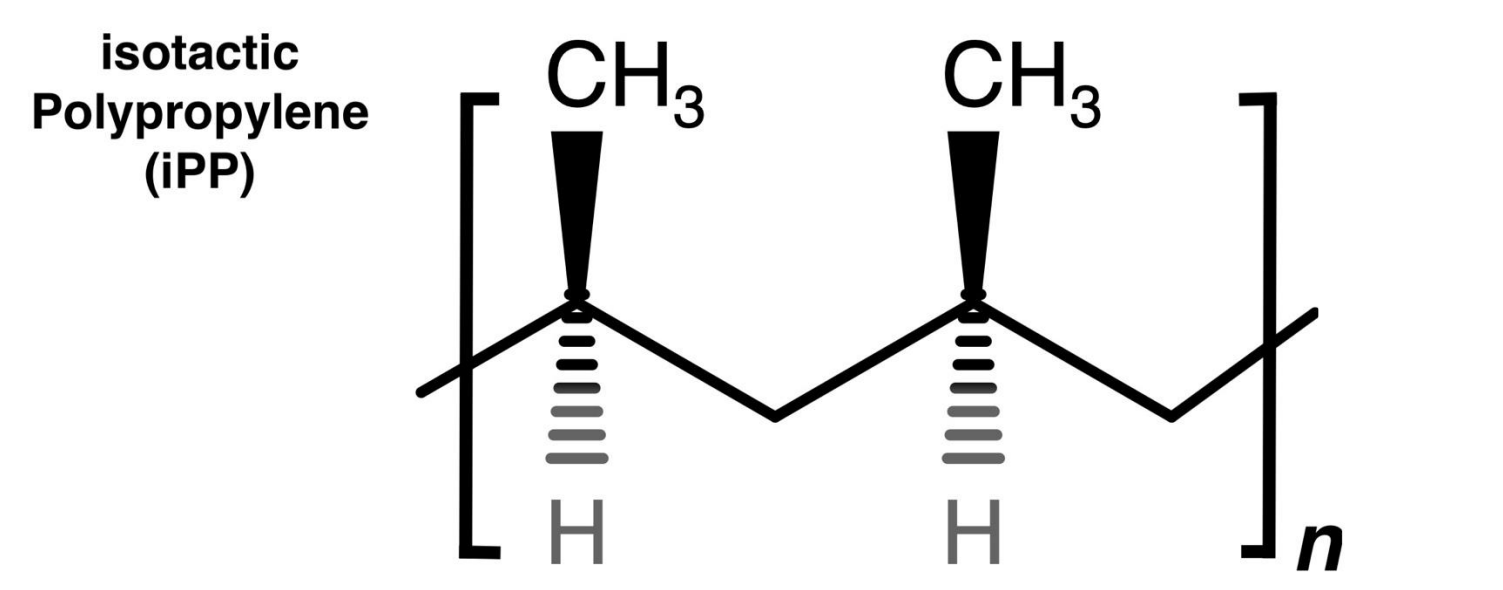
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Introduction

Isotactic polypropylene (iPP) is an important commodity thermoplastic material widely applied in various areas due to its versatility, good physical and mechanical properties, recyclability and low cost. The α - and β -phases are the most common and technologically interesting crystalline forms of iPP. The α -phase with monoclinic crystal structure is thermodynamically the most stable one. In contrast, the β -phase with trigonal crystal structure is obtained almost only by specific nucleation using nucleating agents. [1]



Objective

The aim of this work is studying the correlation between morphological characteristics and structure/composition identity of iPP crystallized in the presence of alkaline earth metal pimelates as nucleating agents.

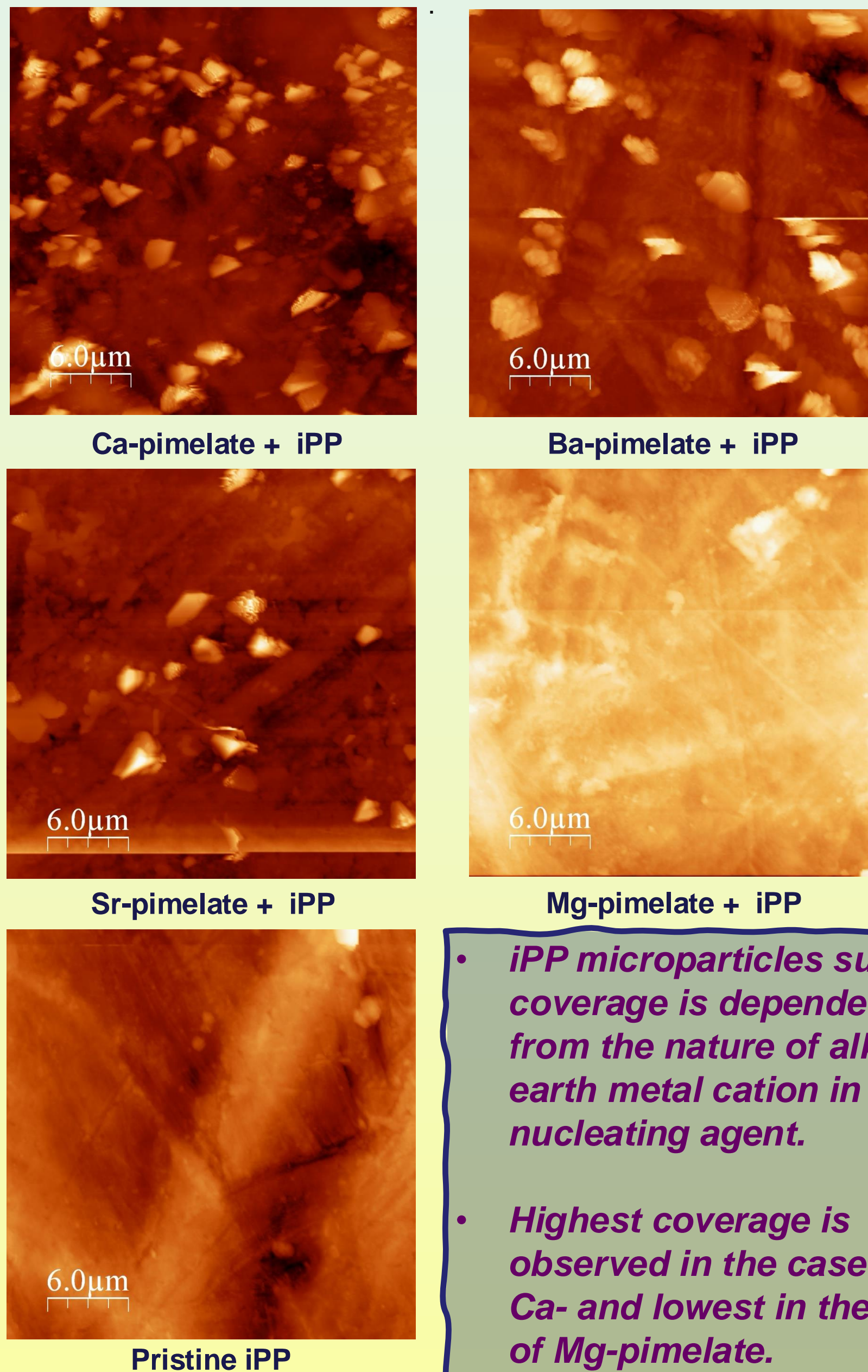
Experimental

Nucleating agent crystallized iPP samples were obtained by mixing 0.3 wt.% alkaline earth metal (Ca, Ba, Sr, Mg) pimelates with iPP in Brabender PL 2000 at 460 K. The samples were heated to 478 K and held in the molten state for 5 min. The crystallization was carried out in the range between 397 K to 406 K.

The morphological characteristics of iPP, crystallized with alkaline earth metal nucleating agents were studied by scanning probe microscopy (SPM), while the chemical and phase identity were identified using Raman spectroscopy and x-ray diffraction (XRD), respectively.

Results

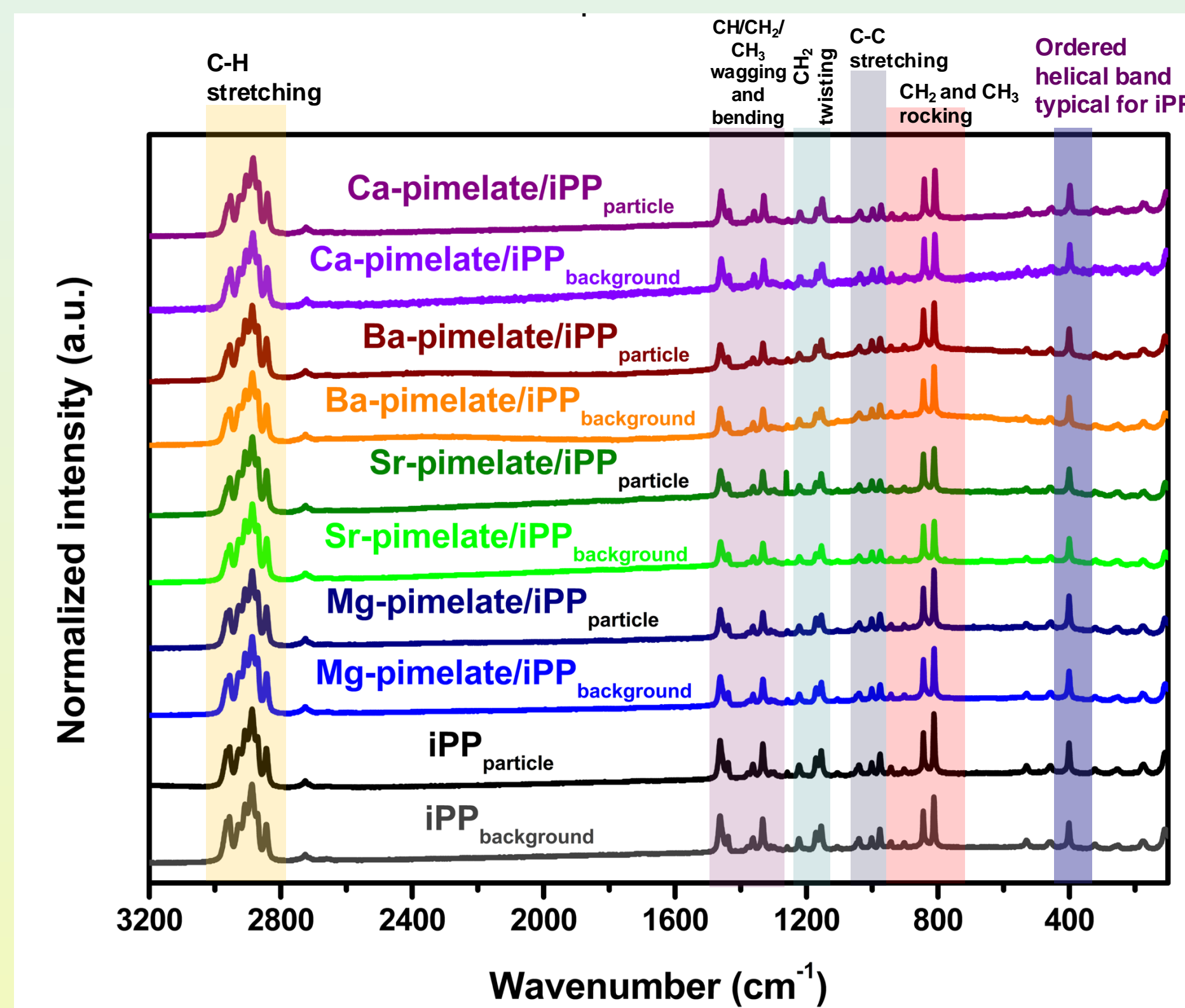
Scanning probe microscopy - SPM



- iPP microparticles surface coverage is dependent from the nature of alkaline earth metal cation in the nucleating agent.*
- Highest coverage is observed in the case of Ca- and lowest in the case of Mg-pimelate.*

Nucleating agent + iPP	Surface coverage of iPP micro particles (number of particles per mm ²)
Ca-pimelate + iPP	1-2 x 10 ⁵
Ba-pimelate + iPP	5 x 10 ⁴
Sr-pimelate + iPP	3 x 10 ⁴
Mg-pimelate + iPP	1-2 x 10 ⁴
Pristine iPP	2 x 10 ⁴

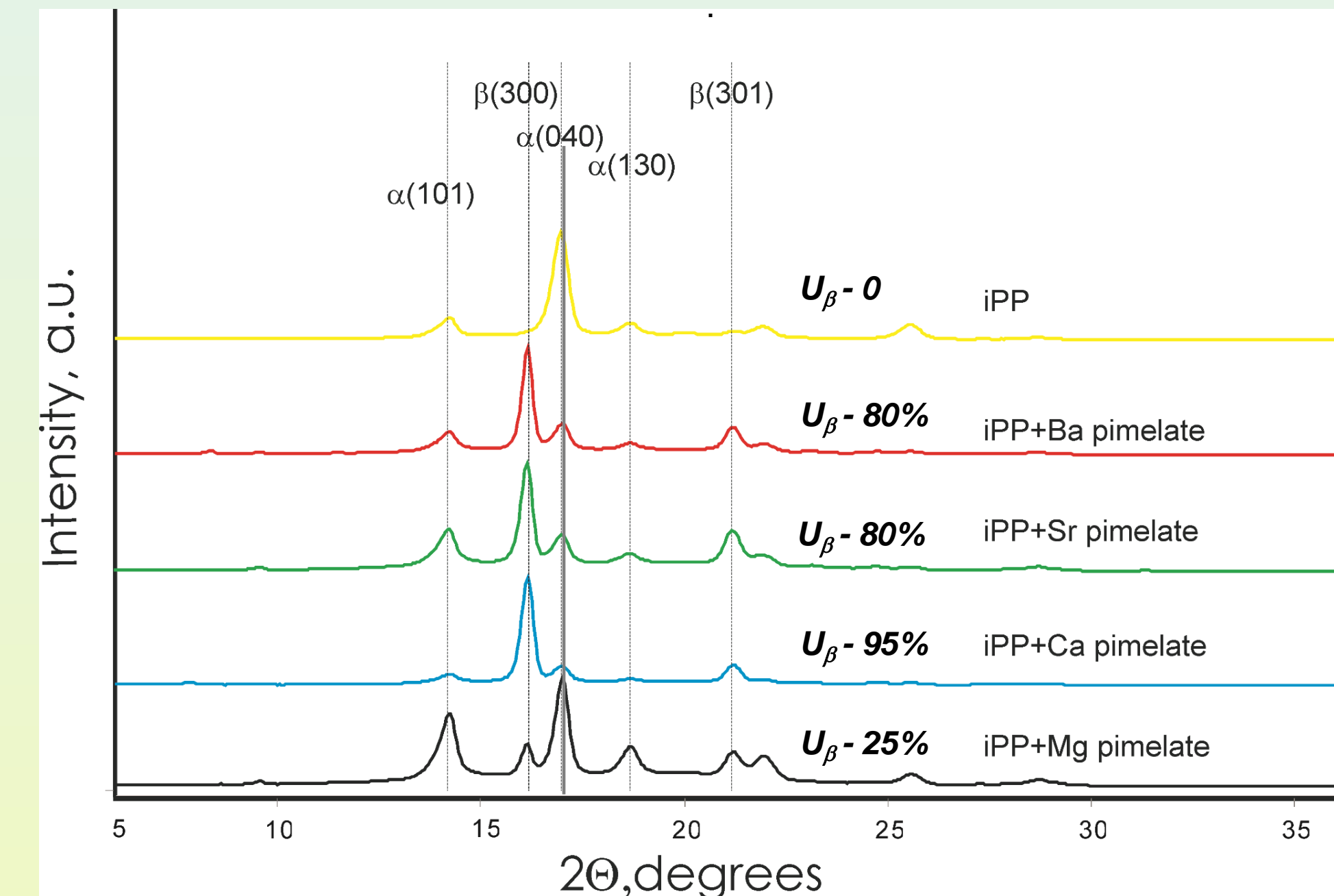
Raman spectroscopy



Particle: Red laser probing a microparticle
Background: Red laser probing the background surface on which the microparticles are scattered

- All Raman spectra show features that are generally typical for polypropylene and specifically for iPP (ordered helical band at 400 cm⁻¹).*
- The polymer chemical composition is not affected by the nature of the nucleating agent cation.*
- The microparticles and background surface share identical chemical composition that corresponds to iPP.*

X-ray diffraction - XRD



- Monoclinic α -phase is practically exclusive in the case of pristine iPP, while the presence of Ba-, Ca- and Sr-pimelate nucleating agents is inducing iPP to crystallize in dominating β -phase (U_{β} = 80-95%).*
- On the other hand, Mg-pimelate induces crystallization in both phases, yet α - is predominating the β -phase. U_{β} for Mg-pimelate nucleated iPP is around 25%.*
- iPP crystallized in β -phase shows higher surface coverage of microparticles, as presented in the SPM images and the table.*
- The highest iPP microparticles surface coverage is achieved when Ca-pimelate is used as a nucleating agent, that is by an order of magnitude higher than all other cases.*

Conclusions

- The utilization of alkaline earth metal pimelate nucleating agents in iPP crystallization affects not only the α - versus β -phase composition outcome, but also the surface coverage with iPP microparticles.*
- The level of microparticles surface coverage is in positive correlation with alkaline earth metal pimelate nucleating agents induced crystallization in β -phase in the following ascending order: Mg, Sr, Ba, Ca-pimelate.*
- This effect is the most prone when using Ca-pimelate as a nucleating agent, while Mg-pimelate favors crystallization in dominating α -phase with the lowest microparticles surface coverage as in the case of pristine iPP that is crystallized without any nucleating agent.*

References

[1] Janevski, A.; Bogoeva-Gaceva, G.; Stefov, V.; Najdoski, M. The Correlation between Structure and β -nucleation Efficiency of Ba, Sr, Ca and Mg Pimelates in Isotactic Polypropylene, *Maced. J. Chem. Chem. Eng.* 2015, 34(1), 189-199. <https://doi.org/10.20450/mjccce.2015.635>

