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April 24 - 27, 2012
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Book of Abstracts

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Advanced characterization techniques
Conductive polymers
Mathematical modelling of processes and properties
Nanofillers: carbon nanotubes, graphene and nanofibres
Nanostructured coatings and adhesives
Polymer materials from renewable resources
Polymer nanocomposites for biomedical application
Polymer networks and gels
Stimuli responsive polymers for optoelectronics, sensing and actuators

Tuesday, April 24

17:00 - 20:00	Registration with Welcome Buffet

Wednesday, April 25

08:00 - 09:00	Registration		
09:00 - 09:10	Opening		
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10:50 - 11:20	Coffee Break		

	Room A Chairperson: R. Turcu	Room B Chairperson: G. Michler	Room C Chairperson: P. Pötschke
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15:50	Coffee and Farewell		

INVERSE-SUSPENSION PRODUCTION OF HYDROGELS: KINETIC MODELLING AND PRODUCT CHARACTERIZATION

Miguel Gonçalves[a], Virgínia Pinto[a], Rolando Dias[a,*] and Mário Rui Costa[b,]**

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Introduction

Hydrogels find important applications in drug and gene delivery, tissue engineering, bio-separations and different fields of biomedicine.[1] End-use properties of such polymer networks are dependent on their structural topology which is affected by the production process used. Formation of non-ideal networks due to reduced reactivity of pendant double bonds and/or cyclization are some mechanisms hindering the prediction of physical properties of hydrogels from their synthesis conditions, which is an important open problem in this research field. This work reports the synthesis/characterization of different classes of hydrogels and also the kinetic modeling of their formation process. Time evolution in batch reactor of key properties of the gelation process was measured, namely monomer conversion, weight fraction of gel, molecular weights and average radius of gyration of the soluble phase. The performance of the resulting hydrogels was also assessed (e.g. swelling ratio in different environmental conditions) in order to link the synthesis conditions with the end-use properties of such materials. A general kinetic approach is used to predict the dynamics of hydrogel formation. Comparison between experimental measurements and these predictions allows the build-up of a computational tool useful in the design of processes for production of tailored hydrogels.

Materials and Methods

Acrylamide (Am) and acrylic acid (AA) were used as the main water soluble monomers. Methylene bisacrylamide (MBAm), trimethylolpropane triacrylate (TMPTA) and 1,6-hexanediol diacrylate (HDDA) were considered as crosslinkers. The influence of the kind of crosslinker (e.g. bifunctional/trifunctional) on the gelation process was also here studied. The pair ammonium persulfate (APS)/tetramethylethyleneamine (TEMED) was used for redox initiation at low polymerization temperature (e.g. 20 °C) as well as 2,2'-azobis(2-methylproprionamidine) dihydrochloride (V50) as thermal initiator (polymerizations at 50 °C). Cyclohexane was chosen as the continuous phase in the inverse-suspension polymerizations with Span 80 as stabilizer. Sodium hydroxide was used in the partial neutralization of acrylic acid and thioglycolic acid as chain transfer agent (CTA). A size exclusion chromatography (SEC) apparatus running with water as eluent, coupled to refractive index (RI) and multi-angle laser light-scattering (MALLS) detectors, was used to characterize the polymer soluble phase. A few solution polymerizations were *in-line* monitored using an immersion probe with FTIR-ATR detection.

Experimental

Polymerizations were performed in batch reactor using the inverse-suspension process. This technique is especially useful for gel production at nearly isothermal conditions and for keeping a good agitation of the reaction vessel. Reactions were carried out at low scale in a 200 mL reaction vessel and at higher scale in a 2.5 dm^3 reaction apparatus.[2] Stable suspensions were produced with a volumetric ratio aqueous/organic phases 0.2, 1% (w/w) of surfactant in the continuous phase and agitation speed at 300 rpm. Argon blanketing was used to prevent inhibition of the polymerization by dissolved oxygen. At prescribed polymerization times, reaction samples were collected from the reactor, quenched at low temperature in a solution containing hydroquinone to stop the reactions, and afterwards prepared to inject the soluble polymer in the SEC/RI/MALLS system. Dynamics of monomer depletion, weight fraction of gel, molecular weights and average radius of gyration were thus measured. Final products, after purification, were characterized in terms of their swelling ratio in aqueous solution.

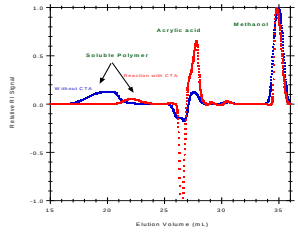
Kinetic Modelling

A general kinetic approach based on population balances of generating functions[3-5] allows the description of gel formation based on the underlying reaction steps. Note that radical crosslinking polymerizations are kinetically controlled and therefore other gelation theories, such as the theory of branching processes, are not

strictly valid for these systems. Kinetic schemes with flexible degree of complexity can be considered in the framework of the present approach, including not only crosslinking mechanisms but also transfers to polymer if needed.[6] Predictions of key properties of non-linear polymers are thus possible before and after gelation.

Results and Discussion

Experimental and theoretical studies include two copolymerization systems (acrylamide/crosslinker and acrylic acid/crosslinker) and one terpolymerization (acrylamide/acrylic acid/crosslinker). The former classes of hydrogels are useful as super-absorbents and the latter as pH-sensitive materials. Figures 1-3 show typical results obtained with the SEC/RI/MALLS anaysis of the soluble phase of hydrogels.



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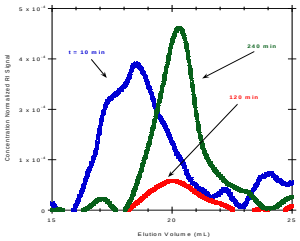

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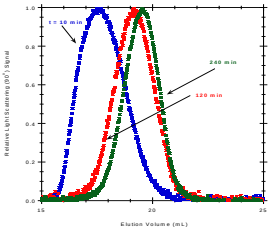

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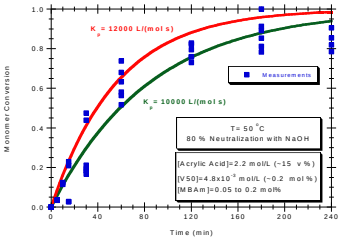

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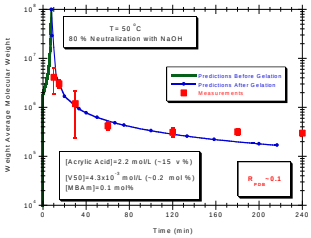

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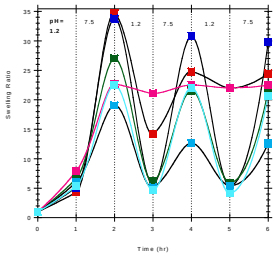

(6)

Typical chromatograms observed with SEC/RI/MALLS characterization of the soluble phase in hydrogels formation (1). Time-evolution of the RI (2) and LS (3) signals of the soluble phase during hydrogels synthesis. Predicted and measured time-evolution of monomer conversion (4) and molecular weight of the soluble phase (5) during hydrogels formation. Repeated swelling (pH=7.5) and collapsing (pH=1.2) of synthesized acrylamide/acrylic acid hydrogels showing their usefulness as pH sensitive materials (6).

Time-evolution of monomer conversion typically observed in the synthesis process is illustrated in Fig. 4 and compared with model predictions. The strong dependence of apparent rate parameters (e.g. k_p of AA) on operation conditions besides temperature (e.g. neutralization and concentration) is an important issue. Time-evolution of the measured molecular weight of the soluble phase during AA/MBAm hydrogel formation is showed in Figure 5 and compared with predictions. Ability of the present kinetic approach to deal with gelation is here illustrated. Special properties of the produced hydrogels are illustrated in Fig. 6, namely the pH-sensitivity (with applications in drug delivery) of Am/AA/MBAm hydrogels synthetized in this work.

Conclusions

Dynamics of hydrogels formation was investigated through their production in batch reactor operating with inverse-suspension. A general kinetic approach was applied in the modelling studies concerning the synthesis of these materials. These results can be used to design the production of tailored hydrogels. Usefulness of the synthesized hydrogels as super-absorbents and pH-sensitive materials was also shown.

Acknowledgements.

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