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MECHANICAL CHARACTERIZATION OF POLYMERIC MICROSPHERES THROUGH MICRO-COMPRESSION TESTING

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Introduction

Usage of polymeric microspheres in biomedical applications is dramatically increasing in the last years. In this study the mechanical properties of a new design of polymeric microspheres to be used as carriers for local release of drugs and grow factors, are investigated. Technically, nanoindentation is applied on microspheres of size in the range 30-70 µm. To measured load-displacement data, the Hertz model is applied to estimate the Young's modulus.

Materials and Methods

The water-in-oil emulsion method was applied to gelatin type A to produce three types of microspheres characterized by a different percentage of genipin. Genipin in three different values of concentration (w/v) %: 0.1% (group 1) ,0.5% (group 2), and 1% (group 3), was used as natural cross-linker.

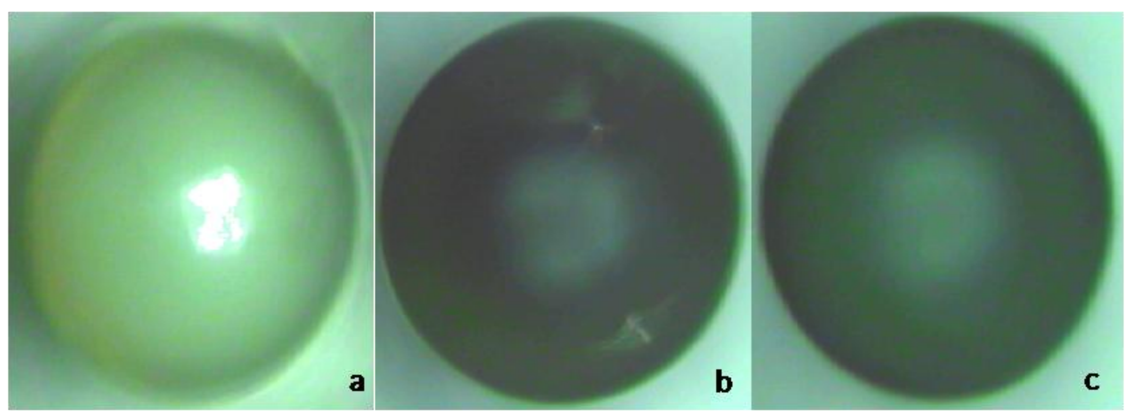


Figure 1 Explanatory images (by optical microscopy) of microspheres:(a) group 1; (b) group 2; (c) group 3.

The Nanoindenter XP (Agilent/MTS company) was adopted and a specific protocol was developed.

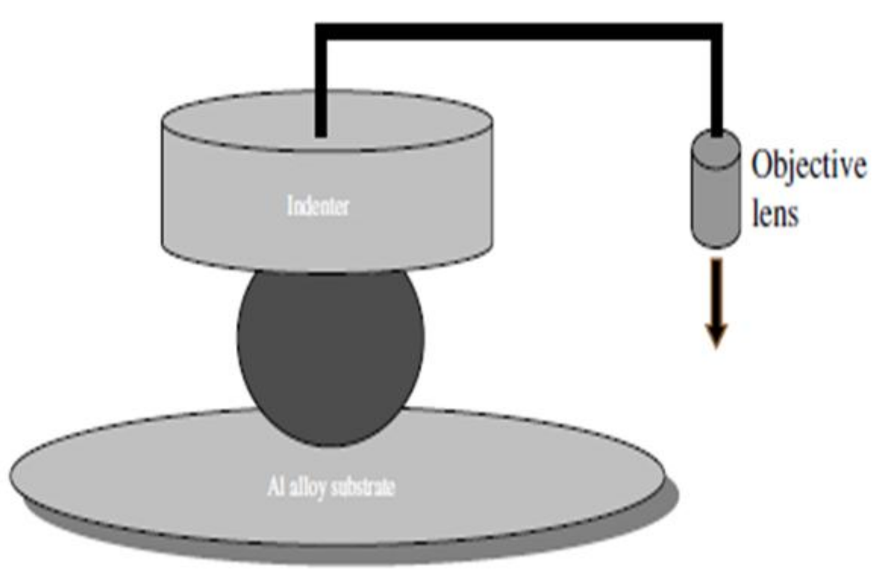


Figure 2 Schematization of the micro-compression test.

A flat end punch with a diameter of 500 µm was selected for indentation. The compression test is characterized by three steps:

- loading, displacement rate was set at a value of 10 nm s-1;
- hold, stabilization period during which the load was maintained for a period of 5 s;
- unloading, same displacement rate of the loading step;
- maximum indentation depth equal to the 5% of the initial radius of the microsphere;

On measured load-displacement curves as obtained from the compression tests, the Hertz model [1] was applied and the Young's modulus was estimated according to:

$$F = \frac{4\sqrt{R}}{3}\frac{E}{1-\upsilon^2}\sqrt{\left(\frac{H}{2}\right)^3} = \frac{4\sqrt{R}}{3}E^*\sqrt{\left(\frac{H}{2}\right)^3}$$

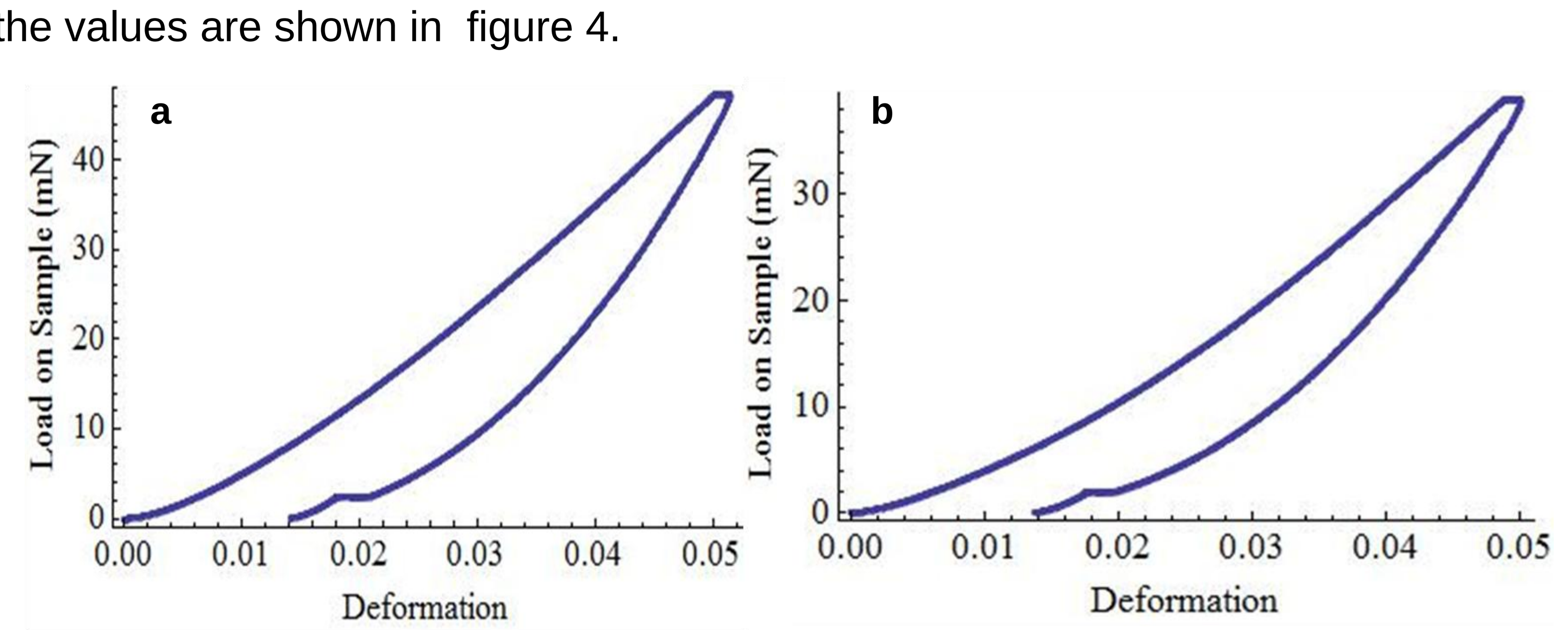
The value of the Poisson's ratio was assumed to be equal to 0.5 [2][3].

Statistical analysis

An analysis of variance (ANOVA) was performed to analyze the influence of the genipin on mechanical properties of microspheres.

Results and Discussions

Typical compression load-displacement curves as obtained from nanoindentation are presented in Figure 3. The Young's modulus was estimated by using the Hertz contact model and the values are shown in figure 4.



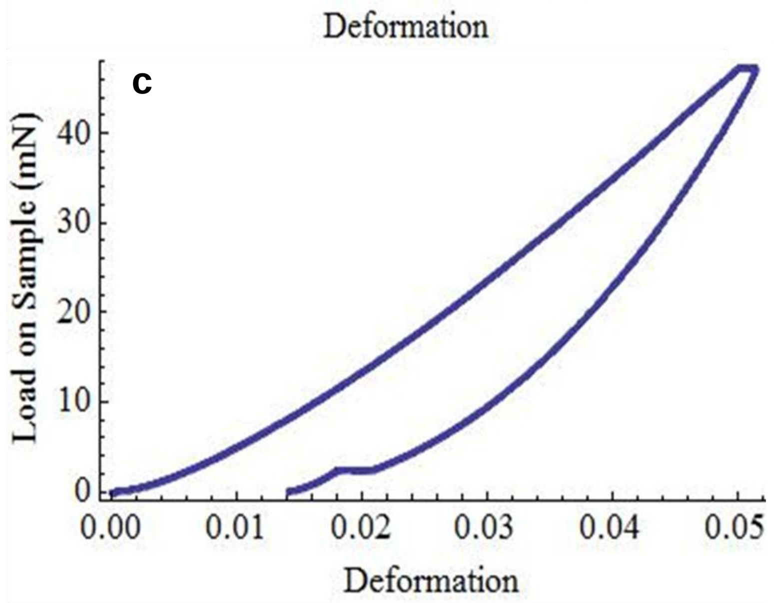


Figure 3 Typical Load-Displacement curves as obtained from nanoindentation tests on gel microspheres: (a) group 1; (b) group 2; (c) group 3. The curves are characterized by an hysteresis loop typically characterizing viscous-elastic materials

The explanatory results displayed in Figure 5 are representative of all the nanoindentation tests performed on single microspheres and indicate that there is a satisfactory agreement between the Hertz model for contact and the experimental data.

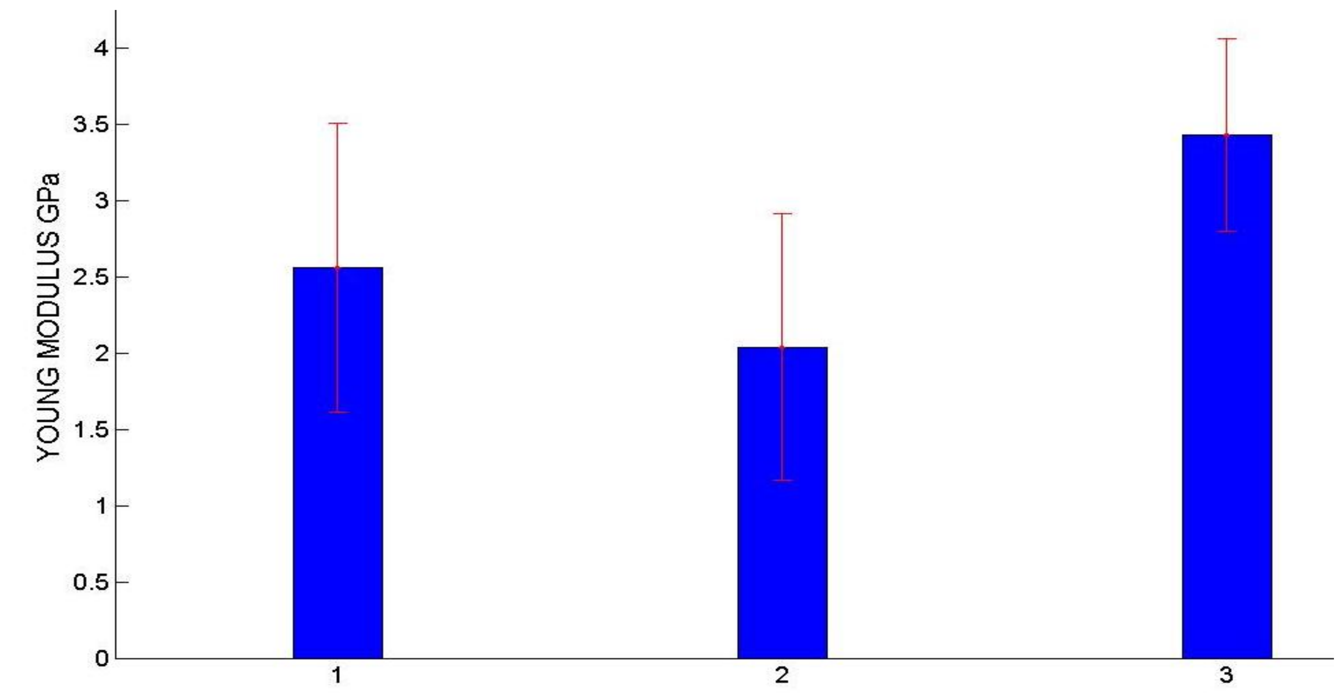
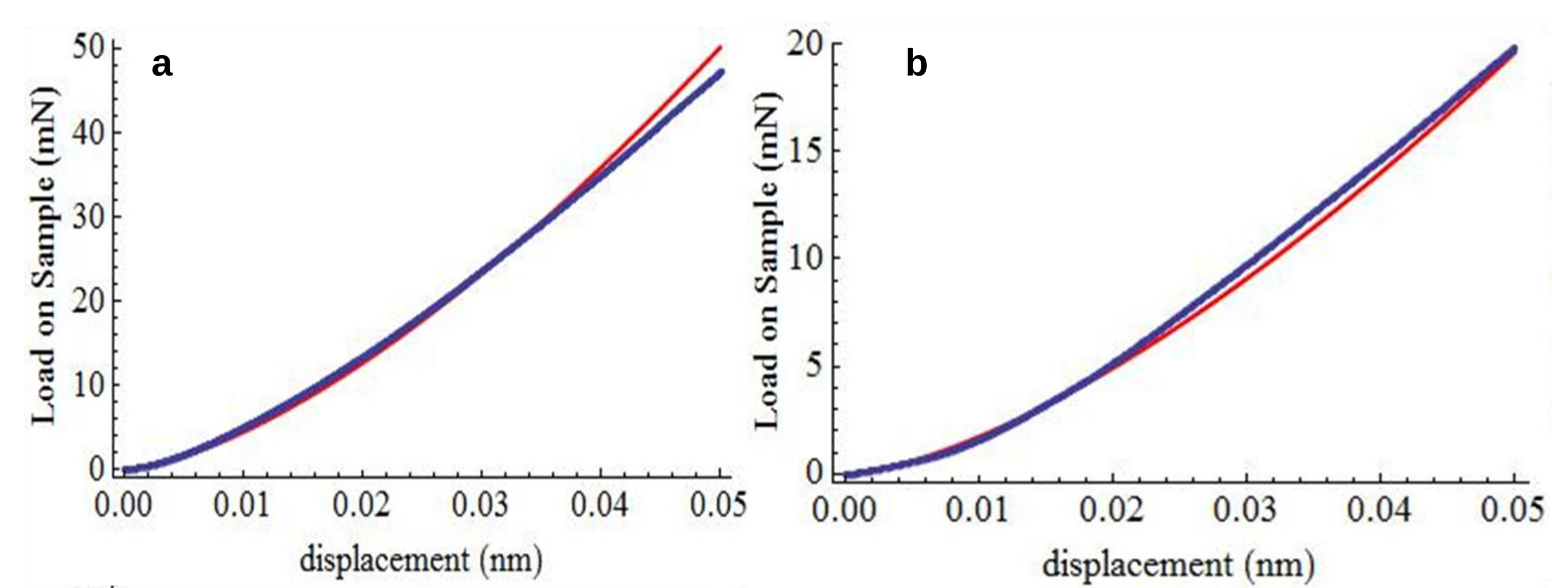


Figure 4 The histogram represent the averaged values of Elastic Modulus as obtained applying the Hertz model.

The percentage of genipin influences the mechanical properties of the microspheres. The analysis of variance (ANOVA) applied to the tests carried out on microspheres shows a correlation ($p<0.05$) between their mechanical properties and the percentage of genipin used during the production of the microspheres. The progressive increase in the percentage of genipin in microspheres composition is not related to a linear increase of the Young's modulus. Furthermore the analysis shows that there is not statistical difference between microspheres of group 1 and 2.



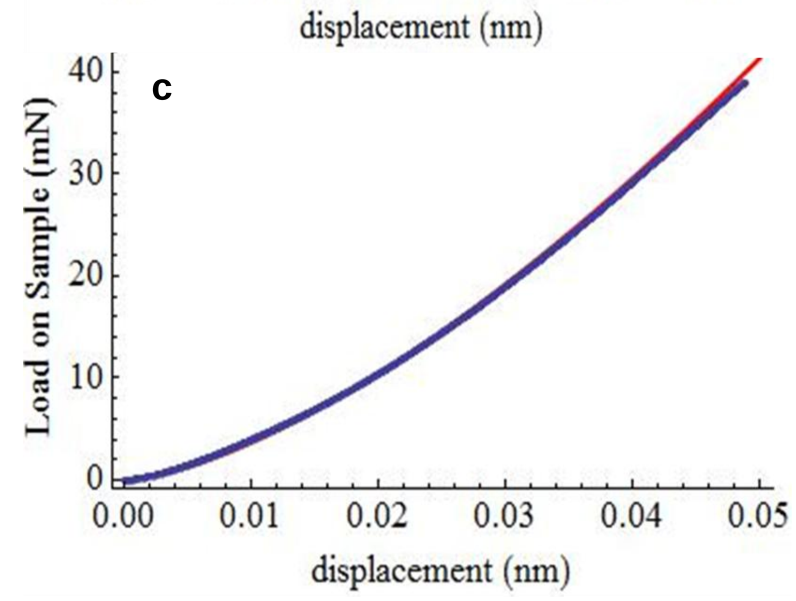


Figure 5 Comparison of the loading phase as obtained from the experimental data (blue curve) and the Hertz model (red curve): (a) group 1; (b) group 2; (c) group 3.

Conclusion

1. The average values of the standard error (and of R^2) with respect to the load-displacement curves estimated by applying the Hertz contact model are equal to 1.55 mN (R^2= 0.98), 1.59 mN (R^2= 0.99), and 2.10 mN (R^2= 0.99) , for group 1 group 2 and group 3, respectively. These data confirm that the selected Hertz model satisfactorily fits the experimental data as obtained from the application of a nanoindentation strategy, making the estimated mechanical properties reliable.
2. The value of Young's modulus for microspheres belonging to group 2 is lower than group 1, so an increasing of genipin of only the 0.4% (w/v) is not able to improve the mechanical characteristics of microspheres.

Several limitations could weaken the findings of this study. In particular, the defined deformation at 5% of the initial radius, which limits the validity of these results only for small deformation. Moreover, nanoindentation tests were carried out in dry condition.

References

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