Proceedings of the 2012 IEEE 12th International Conference on Bioinformatics & Bioengineering (BIBE), Larnaca, Cyprus, 11-13 November 2012

Biodegradable Encapsulation for Inductively Measured Resonance Circuit

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Abstract— **The feasibility of biodegradable encapsulation for LC resonance circuits is studied. The used biodegradable polymers are polycaprolactone (PCL) and poly-L-lactide/caprolactone (PLCL). The encapsulated circuits are immersed in a phosphate buffer solution and the phase and magnitude responses are measured by using an inductive link during an 80-day test period. The features derived from the resonance curves are extracted and studied. The features change fast when the encapsulation absorbs water during the first days of immersion. After the initial water intake, there is a drift in the extracted estimates for the resonance frequency. The drift of the frequency of the resonance circuit in PLCL is faster compared with the drift of a circuit in PCL. The resonance curve of the PLCL specimen also diminished to undetectable after 72 days of immersion. The resonance curves of the sample in PCL were easily detectable throughout the test period. The achieved results promote further studies based on this concept in order to monitor biodegradable polymers and their properties.**

Keywords— ***encapsulation; passive resonance sensor, biodegradable polymer***

I. INTRODUCTION

Passive resonance sensors are LC circuits which are measured via inductive coupling between the sensor and a reader coil. The measurand affects the resonance frequency of the circuit due the inductance or capacitance. There are also configurations in which the resistive component (dissipation) of the resonance circuit is measured. The passive resonance sensors have been tested for measuring pressure [1], biopotential signals [2], chemical [3] and even biological variables [4]. This method has also been used to detect moisture [5]. The advantages of this measuring method are the simple and small structure of the actual sensor and the possibility to make wireless measurements through the non-conductive medium. The structurally simple sensor eases the developing process when new materials and methods are tested. These two advantages of the passive resonance sensors in combination, justify an effort of developing a totally biodegradable sensor based on the concept of passive resonance sensing. Experimental resonance circuits have been made of biodegradable metals and polymers [6]. However, it is still a challenge to make these devices to function reliably in an aqueous environment.

There are several types of biodegradable polymers, typically classified into two groups: synthetic ones and those derived from natural resources. The polymers have different properties, for example degradation rate can vary from hours or days to months and even years. Another classification for the biodegradable polymers is the degradation type; they can degrade via surface erosion or via bulk erosion. Water penetration and diffusion into the polymer structure are the main reasons for which type of degradation behavior polymer shows. In surface eroding polymers, water diffuses into the structure very slowly and degradation occurs in a layer near the surface. On the other hand, in bulk eroding polymers, water diffuses completely into the device thus degrading the polymer throughout [7].

The resonance behavior of the open LC circuits is prone to environmental effects, mainly due to the changes in the permittivity and conductivity of the environment close to the circuit. The permittivity affects due to the parasitic capacitances and shifts the found resonance frequency. The conductivity will lead to losses which show as reduced Q-factor of the resonator thus reducing the useful range of in which resonance can be detected. In order to reduce the effect of the environment, the circuit can be encapsulated with a dielectric material. If the material is biodegradable, the circuit can be used to study the properties of the polymers and composites which change by the water diffusion and degradation.

In this paper, the effects of the biodegradable encapsulation on the properties of the resonance circuit in aqueous environment are studied. The subject is relevant because the degradation processes of these polymers involve interaction with water and many polymers actually absorb water. There is a need for an unobtrusive method to monitoring the degradation process of the polymer in the aqueous environment. The first aim is to verify the feasibility of the biodegradable encapsulation for a LC resonance circuit and the used wireless measurement method in general. Next, we study if there are reliably extractable features that can be used to

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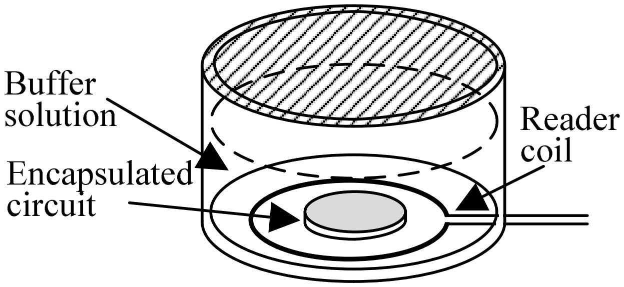

Figure1. Measurement set-up.

monitor the degradation or water absorption processes in the materials.

II. METHODS

A. Measurement set-up

In this work, the resonance behavior of LC circuits encapsulated into a biodegradable polymer was studied. The behavior was measured wirelessly by inductive coupling while the circuit was in a buffer solution inside a closed cell culture well (Fig. 1). The measurements were done with a portable reader device [8]. The impedance phase and magnitude values were swept at discrete frequencies over the range of 10 MHz. The frequency resolution of the measured data points was 100 kHz. The used Sörensen buffer solution is one of the commonly used mediums when studying the biodegradable devices and the buffering prevents the solution to turn acidic due to the degradation products of the polymer. In the measurement set-up, the encapsulated resonance circuit is submerged in 10 ml of the buffer solution in a commercial plastic 6-well cell culture plate (in 37 °C). The phase and magnitude responses of the sensor were measured by a flat reader coil (diameter of 40 mm) which was placed under the cell culture plate just below the circuit. In this setup, the encapsulated circuit was at the bottom of the well but the exact location of the sensor device could slightly vary.

B. Resonance Circuit

In this study, we used the LC circuits made of non-degradable materials inside the biodegradable enclosure. The coils are made of insulated copper wire (thickness 0.17 mm, round cross-section). The coils have the diameter of 10 mm and they have 3 turns. A surface mounted capacitor (0603 casing, 22 pF capacitance) is soldered inside the coil to complete the LC circuit. The geometry of the LC circuit affects its sensitivity to environment due the parasitic components, mainly due the parasitic capacitances. The method and the used materials were selected as such to get as simple and easy-to-make circuit as possible. A small bulk capacitor was selected instead of parallel plane configurations as it is less sensitive to the environmental changes. This keeps the changes of the resonance in a measurable range. In this case, the coil is insulated but the soldering and the contacts of the capacitor are prone to environment. This is intended to be the point where the circuit breaks when the encapsulation is dissolved.

C. Biodegradable Encapsulation

The resonance circuits are encapsulated into two different biodegradable polymers: polycaprolactone (PCL) homopolymer and PLCL (70/30 L-lactide/caprolactone) copolymer. They were selected due to their relatively mild melt-processing temperatures (low glass transition temperatures). In the encapsulation procedure (compression molding), the polymer granules are first vacuum-dried and compressed into sheets of specific thickness using the combination of temperature and pressure. The circuit was then encapsulated between two sheets and compressed to a predefined thickness by using heat to seal the polymer and to embed the component into the polymer. Finally, the polymer is cut to disk shape (diameter of 22 mm). A control circuit of similar shape was prepared by using biostabile polydimethylsiloxane (PDMS) for an encapsulation polymer.

D. Signal Prosessing

We study if there are features of resonance which can be reliably extracted from the measured data. A feature related to the resonant frequency of the circuit is needed. In addition, we want to extract features that depend on the Q-value of the circuit. In the ideal case of a passive resonance sensor, these features would be independent of the measurement environment and the coupling between the used coils. In this study, however, the environment close to the circuit affects the extracted features and we use this effect to monitor the degradation of the encapsulation.

The measured data contains the phase and magnitude values of impedance at discrete frequencies. First the baseline is removed. Then the features are extracted from the data by fitting a polynomial to the data and evaluating either the maximum or minimum. In the case of the phase response the extracted features are the frequencies of the maximum and minimum values of the resonance curve. In addition, the height and width of the curve are evaluated. The height is the absolute phase value between the maximum and the minimum. The width is the difference of the frequencies. In the case of magnitude, the frequency of the minimum of the magnitude dip was extracted. The polynomial fitting is used to increase the resolution of the extracted features. Finally the calculated features were averaged. Each data point presented in the result section is an average of 100 samples.

III. RESULTS

A. Feasibility of the Biodegradable Encapsulation

PCL gave the better quality encapsulation for the resonance circuit. Due to the relatively good water diffusion coefficient of the semicrystalline and hydrophobic PCL, it seemed to be a good candidate for the encapsulation of sensors because of its fast stabilization in the aqueous environment. The diffusion coefficient of PCL is higher than that of the other biodegradable polymers, such as PLLA (poly-L-lactide), PHB (polyhydroxybutyrate) and PGA (polyglycolide). The crystallinity of PCL influences the water diffusivity [9]. The PLCL (70/30 L-lactide/caprolactone copolymer) material was also tested, however, it needs higher processing temperature and thus the encapsulation was more unreliable due to the

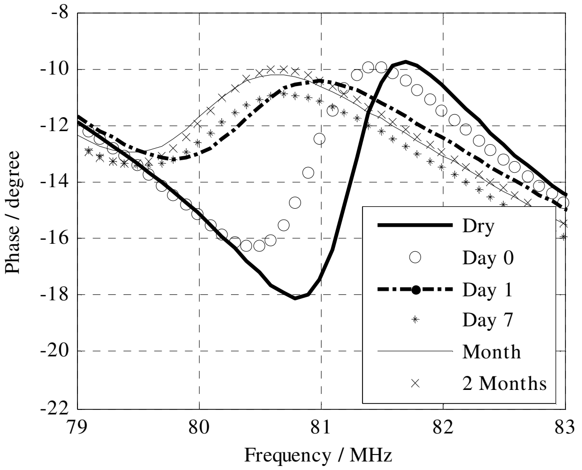

Figure 2. The measured impedance phase curves of the PCL encapsulated circuit.

limitations in processing. Some of the test circuits were damaged and the overall encapsulation was more irregular compared with PCL.

B. *Characteristics of Measured Data*

The encapsulated resonance circuits were measured wirelessly. An example of the impedance phase measurement made with a circuit encapsulated in PCL is illustrated in Fig. 2. When the circuit was submerged (day 0), the resonance induced curve in the phase response was shifted to the lower frequencies and the height of the pattern was decreased slightly. After the day 1, the height of the curve is notably smaller and the pattern is also wider compared with the measurement made with the dry circuit. After the day 1 it is hard to detect significant changes in the shape of the curve. The phase measurements of the circuit in PLCL after immersion are shown in Fig. 3. The resonance curve shifts continuously to lower frequencies and the height of the curve gets smaller. After two months, the curve is hard to detect.

The measured magnitude response of the PCL circuit is shown in Fig. 4. The frequency of the magnitude dip shifts to lower frequencies after immersion in a similar way as in the case the frequency of the maximum of the phase curve. The dip in the magnitude response also gets wider and lower. The baseline of the curves varies between measurements. Note, the form of these example phase and magnitude responses are typical of this set of circuits and used reader coil. The form can vary, especially if the relative inductances of the used coils are changed.

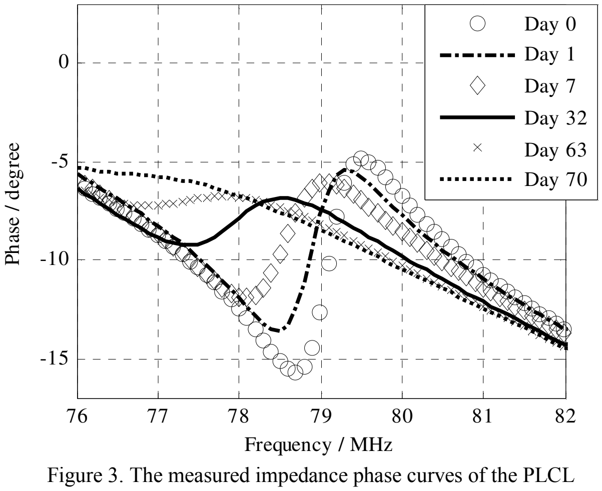

Figure 3. The measured impedance phase curves of the PLCL encapsulated circuit.

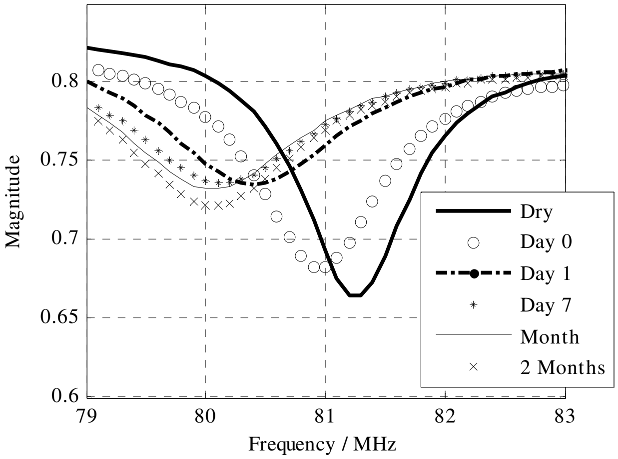

Figure 4. The measured magnitude curves of the PCL encapsulated circuit.

C. *Extracted Features*

In the experimental test series, the features extracted from measurements made with the resonance circuits encapsulated in PCL, PLCL and PDMS are studied. The shifts of the resonance frequency estimates derived from the maxima of the phase curves are shown in Fig. 5. According to the data, the frequency shift due to the immersion is clearly visible. In the case of PCL, the shift of frequency continues rapidly for the first five days. After that period, only a slight drift remains. The initial drop of the frequency is bigger in the case of PCL compared with PLCL. After the initial drop, the frequency of

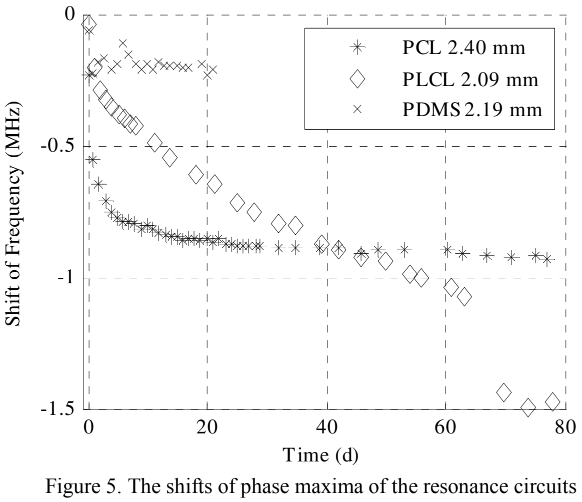

Figure 5. The shifts of phase maxima of the resonance circuits encapsulated in PCL, PLCL and PDMS after submersion to buffer solution.

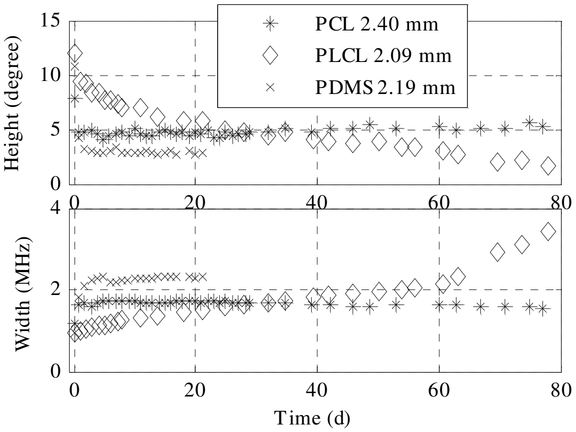

Figure 6. The height and width of the measured the resonance curves (impedance phase).

the PLCL encapsulated circuit starts to drift faster compared with the PCL encapsulated circuit. After 63 days there is a significant drop in the frequency, yet the resonance features can be extracted for another 15 days. After the day 78 the resonance curve is too small to be detected. The frequency of the sample encapsulated in PDMS shifts rapidly during the first day and then it settles on a rather stabile level. The PDMS encapsulated circuit was immersed and measured only for three weeks.

The heights and widths of the measured phase curves are shown in Fig. 6. Both of these features are dependent on the Q-factor of the resonator and indicate how reliably the circuit can be detected with the wireless method. The high and narrow resonance curve is easier to detect from a long distance. The height is also strongly dependent on the coupling factor between the coils which in this test data is assumed to remain almost constant. In the case of PCL the height of phase curve drops and the width increases during the first day after immersion but they stabilize afterwards. In comparison to PCL, the height of the resonance curve of the sample in PLCL does not stabilize. It drops steadily after the initial water intake. The width of the resonance curve of the circuit in PLCL increases steadily until the day 63 after which the width increases more rapidly. The height of the phase curve of the circuit encapsulated in PDMS drops significantly during the first day. In the case of PDMS, the width of the resonance curve also widened significantly during the first day which partly explains the small shift of frequency of the maximum of the phase curve in the Fig 5.

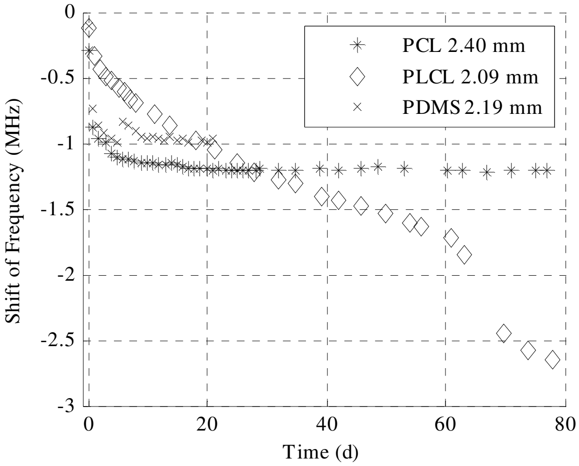

Figure 7. The shift of the frequency of the dip in the magnitude curve.

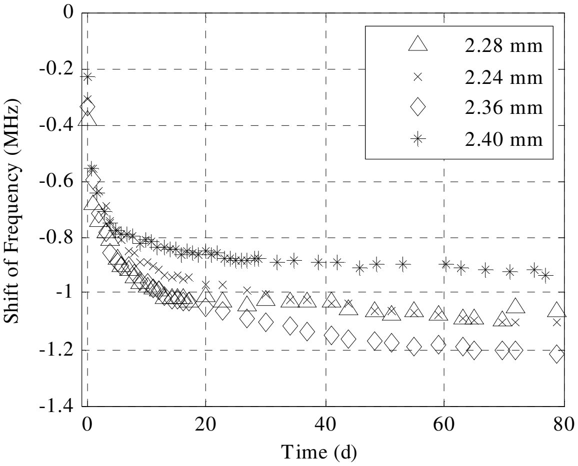

Figure 8. The shifts of the phase maxima of the four resonance circuits encapsulated in PCL and submerged in buffer solution.

The frequencies of the minimum of the magnitude dip are shown in Fig 7. The behavior of this feature is very similar to the feature of the maxima of the phase curves. However, the changes are larger during the transit period after the immersion. Notably, for the PDMS encapsulated circuit the shift of the magnitude dip is much larger compared with the shift in the Fig. 5.

D. Repeatability of Measurements

The repeatability of the presented method was estimated by measuring four similar samples encapsulated in PCL. In the Fig. 8, the shifts of the maxima of the phase curves are shown. The thickness of the encapsulated samples varies from 2.24 mm to 2.40 mm. The overall shape of the frequency shifts is similar. There is a difference in the initial shifts of resonance frequency due to the immersion and the shifts that occur during the following water intake. The rates of the drifts after the water intake period vary between the samples. The 2.4 mm thick sample is the same as the one used in the section C.

E. Stability of Resonance Circuits in PCL

According to the measured data, the PCL encapsulation makes resonance circuit more stabile as a function of time compared with the PLCL encapsulation. To estimate the achieved stability in PCL and to compare extracted resonance frequency features, the frequency shifts after twenty days of immersion were studied. The extracted features from the phase curve maximum and magnitude curve minimum of two samples are illustrated in the Fig. 9. The rates of the drifts vary between the samples. The noise of the measurement is significant in comparison with the drifts. The tested frequency features provide similar information.

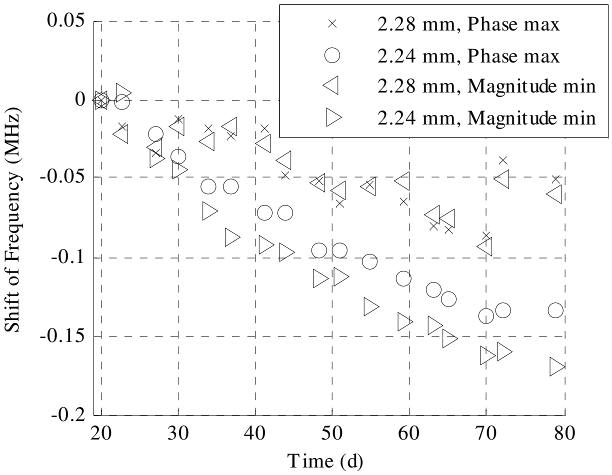

Figure 9. The stability of the phase maximum and the magnitude minimum features of two tested samples in PCL after twenty days of immersion.

DISCUSSION

The biodegradable polymer encapsulated LC resonance circuits were studied. The encapsulation decreased the effect of the environment on the circuit and enabled the inductive measurement of the resonance while the encapsulated circuit was immersed in a buffer solution. The effect of the immersion is still detectable despite the encapsulation. A thicker encapsulation may reduce the initial shift of the resonance because it reduces the change of parasitic capacitances. When the encapsulated circuit was intact, the resonance was clearly detectable and the resonance dependent features were easily extracted immediately after immersion.

The most significant change in the extracted features occurs during the first few days in the immersion. This is probably due to the water absorbed by polymer. The speed and magnitude of the change are governed by the chemistry of the polymer, by the thickness of the encapsulation and by the temperature of the immersion solution. After a few days of immersion, the absorption of water saturates and the polymer cannot take in more water. The absorption of the water will decrease the resonance frequency of the resonator because it increases the parasitic capacitances. Other notable effect is the increase of the losses of the resonator circuit which can be detected as the lowered the height and increased the width of the phase and magnitude curves. The obvious reason for the losses is the increased quantity of the water in the proximity of the circuit but the exact origins of these losses is unclear.

The repeatability of the encapsulation was analyzed with parallel samples. The similar behavior of features was detected with four similar circuits. The initial shifts and the remaining drifts are slightly different but this can be explained with the inaccuracy of the experimental encapsulation process and the variation in the positioning of the coil inside the encapsulation.

The behavior of the extracted features of the resonance circuit encapsulated in PCL stabilizes after the first few days. The estimate of the resonance frequency can be extracted from the measured data with the tolerable uncertainty. This may enable to design passive resonance sensors that measure other variables than the degradation based on the tested concept. However, the remaining drift has to be taken into consideration.

The concept of embedding a resonance circuit inside a biodegradable polymer was found to be useful to investigate the properties of polymers, like water intake. The main advantages of tested PCL in this concept are the low processing temperature and the fast stabilization after the initial submersion. The circuits encapsulated in PLCL may well be good candidates for further studies to show the reliability of the concept due to their faster degradation time. This, however, requires improvement in the processing methods and conditions.

REFERENCES

[1] C. C. Collins, “Miniature Passive Pressure Transensor for Implanting in the Eye,” IEEE Trans. Biomed. Eng., vol 14, pp. 74-83, April 1967

[2] J. Riistama, E. Aittpkallio, J. Verho and J. Lekkala, “Totally passive wireless biopotential measurement sensor by utilizing inductively coupled resonance circuits,” Sens. Actuators, vol. 157, pp. 313–321, February 2010

[3] B. E. Horton, S. Schweitzer, A. J. DeRouin and K. G. Ong, “A varactor-based inductively coupled wireless pH sensor,” IEEE Sensors J., vol. 11, no. 4, pp. 1061-1066, April 2011.

[4] M. S. Mannoor, Hu Tao, J. D. Clayton, A. Sengupta, D. L. Kaplan, R. R. Naik, N. Verma, F. G. Omenetto and M. C. McAlpine, “Graphene-based wireless bacteria detection on tooth enamel, ”Nature Communications, vol 3, March 2012

[5] J. B. Ong, Z. You, J. Mills-Beale, E. L. Tan, B. D. Pereles and K. G. Ong, “A wireless, passive embedded sensor for real time monitoring of water content in civil engineering materials,” IEEE Sensors Journal, vol. 8, issue 12, pp. 2053-2058, Dec. 2008.

[6] C.M. Boutry, H. Chandrahalima, C. Hierolda, “Characterization of RF resonators made of biodegradable materials for biosensing applications,” Procedia Engineering, vol. 25, pp. 1529-1532, 2011.

[7] J.A. Tamada and R. Langer. Erosion kinetics if hydrolytically degradable polymers. Proc. Natl. Acad. Sci, vlo 90, pp. 552-556, 1993.

[8] T. Salpavaara, J. Verho, P. Kumpulainen. and J. Lekkala, “Readout methods for an inductively coupled resonance sensor used in pressure garment application” Sens. Actuators, A, vol. 172, pp. 109-116, Dec.2011.

[9] Jin-San Yoon, Hae-Won Jung, Mal-Nam Kim, Eun-Soo Park, “Diffusion coefficient and equilibrium solubility of water molecules in biodegradable polymers,” J. Appl. Polym. Sci., vol. 77, Iss. 8, pp. 1716–1722, 2000.