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Modification of Polycaprolactone microspheres surface by ultra violet/ozone treatment to improve its hydrophilicity

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Abstract

Unloaded microspheres were prepared from Polycaprolactone (PCL) using an oil-in water emulsion solvent evaporation method. The microspheres with the size distribution in a range of 150-200 µm were then treated by exposed the microspheres under ultra violet irradiation and simultaneously aerated with ozone (UV/O_3) to modify the surface of the beads. The experimental results on surface modification of PCL revealed that the UV/Ozone treatment introduced hydroxyl and carbonyl group on the beads surface and that was confirmed by FTIR analysis and water contact angle. The existence of carboxyl groups grafted on PCL surface was verified quantitatively by absorbance spectroscopy where Toluidine Blue O was employed to react with carboxyl groups to generate an absorbance at 625nm show that the surface of the microspheres had became more hydrophilic.

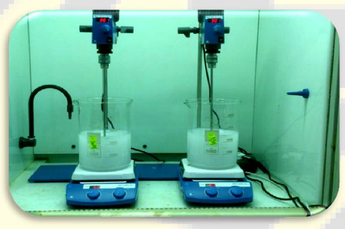
Keywords: microspheres; UV/O_3; Polycprolactone; surface modification

Introduction

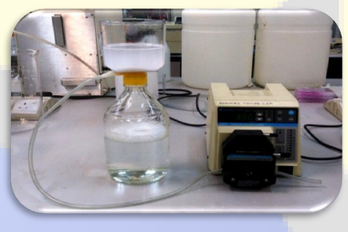
Polycaprolactone PCL will be used in this study because of its potential to be commercialized due to its vast advantages in biomaterial field and tissue engineering, and it's relatively cheaper cost compared to other biomaterials such as dextran and glass. PCL microspheres were fabricated (size range of 150µm - 200µm) by using oil in water (o/w) emulsification coupled with solvent evaporation method. This method is relatively simple, rapid, low-cost and does not involve excessive chemicals and specialized equipment when compared to other options such as seeded polymerization or electro spraying into liquid nitrogen method. Furthermore, self-preparing the microspheres will contribute to lower cost of microspheres production. The surface charge of PCL microspheres could be improved by using ultraviolet/ozone treatment (UV/O_3). Based on theory, charged surface microspheres may facilitate the protein immobilization on the surface and cell adhesion. Treatment using ultraviolet/ozone does not require excessive chemicals and involves relatively easy operation procedures.

Methodology

Oil in water emulsification and solvent evaporation



Vacuum filtration



UV/O_3 treatment

Surface analysis

Result and Discussion

The contact angle of a liquid on a solid surface depends on the physical and chemical properties of surface, such as wettability, hydrophilicity, and roughness. From the result, the contact angle decrease after the treatment with UV/O_3. This can be concludes that the microspheres surface has become more hydrophilic after the treatment indicate that the hydroxyl and carbonyl had been introduce on the surface. The amount of the carbonyl group on the surface of the microspheres measured shows the increment in the amount of carbonyl group from 643.91nmol/g to 1511.74nmol/g.

Table 1: contact angle and TBO assay result the amount of COOH group before and after the UV/O_3 treatment

Microspheres	Contact angle (°)	COOH concentration (nmol/g)
Raw PCL	84.49	643.91
UV/O_3 PCL	78.80	1511.74

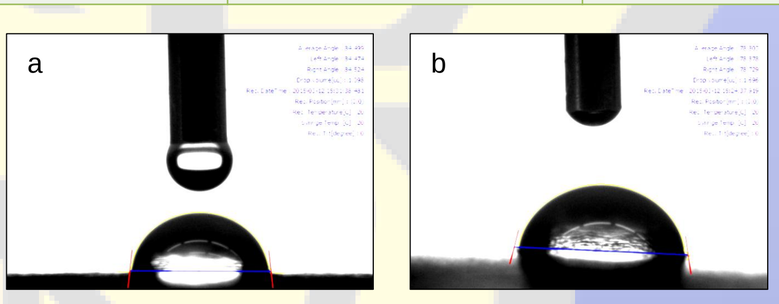
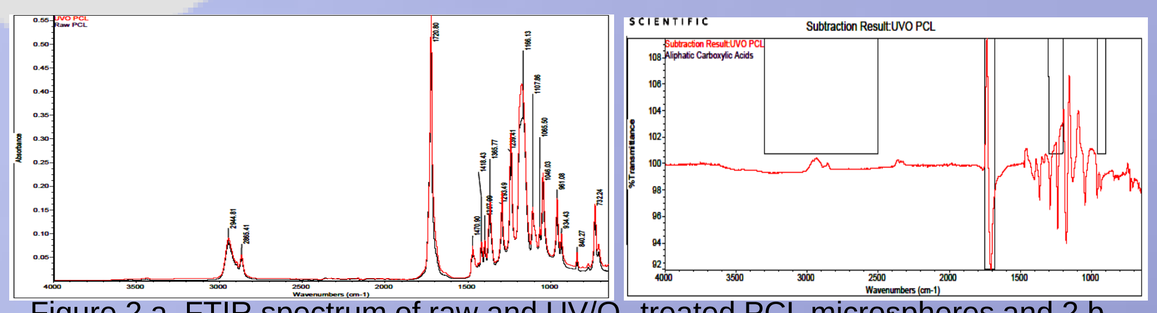


Figure 1: Water contact angle a) before treatment, b) after UV/O_3 treatment

Figure 2 a. shows the result of FTIR of a raw and UV/O_3 treated PCL microspheres. Since PCL backbone also consist of carbonyl functional group, therefore the peak of COOH group introduced during the treatment is not significantly visible. Figure 2 b. is the spectrum shows the subtraction of UV/O_3 to raw PCL microspheres. From this spectrum it can clearly shows the addition of carboxyl functional group on the surface after treatment.



a b

Figure 2 a. FTIR spectrum of raw and UV/O_3 treated PCL microspheres and 2 b. FTIR spectrum of subtraction of UV/O_3 PCL to raw PCL microspheres

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