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Modified Polymer Matrix Nano Biocomposite for Bone Repair and Replacement- Radiological Study

Abstract- Synthetic biomaterials for bone repair and substitute must be yield to critical criteria , one of them, their mechanical properties should not be very less than that for natural bone to prevent fast failure, nor much higher to prevent stress shielding effect which led to fast implant collapse. Other important issue, it should not cause inflammation in implantation region. According to these criteria, many researchers investigated several biomaterials. Titanium dioxide (TiO_2) reinforced polyetheretherketone (PEEK) biocomposite is considered a promising biomaterials, because of their superior properties and good biocompatibility with host tissue. Alumina (Al_2O_3), considered a bio inert ceramic used in this work to modify mechanical properties. Hot pressing technique adopted in this work with pressing pressure of 50 MPa at 370,380,390, and 400 C° compounding temperatures to produce different compositions implants. Animal model used to study inflammation behaviour for implants as a comparison with control group. X-Ray radiological test were carried out for both, implants and control regions. Mechanical testing shows good values, similar to natural bone. No inflammation observed in injury area.

Keywords- Biocomposite, Bone repair, Radiological study, PEEK, Titanium Oxide

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1. Introduction

PEEK is considered one of promising thermoplastic, with extraordinary mechanical properties. The value of Young's modulus of elasticity is 3.6 GPa while its tensile strength is 170 MPa. PEEK has partial crystallinity; it melts at approximately 350 $^\circ C$. PEEK resists thermal degradation and resists to both, organic and aqueous environments [1].

Recently a great interest has taken place in biomedical application for PEEK; especially in bone repair and replacement, due to superior mechanical properties and high thermal and chemical stability [2].

Bone is considered a natural composite, which consists of mineral materials (Hydroxyapatite) and collagen, and just like other composite materials, mechanical properties of bone are highly dependent on both of composition and structure of bone, which includes arrangement of the components, and bonding between fibres and matrix. Arrangement of fibres can differ for several types of bones, and this gives rise to distinct properties [3].

An understanding of mechanical behavior of bone is considered very important to evaluate the risk of fracture. In some types of bones, like vertebrae, which are submitted to compressive loads constantly, there is an increased risk of fracture. Almost all bones are composed of

cortical bone (outer shell), and trabecular bone (an inner bone). The fractures in vertebrae, hip and wrist tend to start the global structural failing process in the regions of trabecular bone with decreased in both of bone mass, and microarchitecture changes [4].

Titanium dioxide (TiO_2) implants have been widely considered as orthopedic materials [5], and Nano TiO_2 have high biocompatibility and bioactivity. The major constrain in orthopedic implantations is local inflammation, which is caused by metal implant debris from wear and corrosion. This issue is considered to be the major cause of bone loss and implant failure; second issue is stress shielding because of mismatch between implant material and bone tissue [6].

An alumina ceramic is considered as bioinert, and has characteristics of high hardness and high abrasion resistance. These excellent wear and friction behavior of Al_2O_3 is related to both of surface energy and surface smoothness of this ceramic. Thermodynamically, there is only one stable phase [7,8].

This study investigates the effect of adding Nano Al_2O_3 particles on the mechanical properties and inflammation behavior of Nano TiO_2 /PEEK biocomposite using animal model.

2. Experimental Procedure

I. Synthesis of bio-composite materials

PEEK powder have an average particle size of 10 μ m, with a nominal density of 1.3 g/cm³ supplied by Right Fortune Industrial Limited (Shanghai, China) were used as polymeric matrix. TiO₂ and Al₂O₃ were used as ceramic fillers; TiO₂ (99% pure) having 40 nm average particle size and a 4.23 g/cm³ particle density, while α -alumina powder has an average particle size of 10nm and a density of (3.890 g/cm³). Both ceramic powders were supplied by M.K. Nano (Toronto, Canada). Ball mill mixing used to mix powders with different compositions: 10 vol%TiO₂/PEEK, 20 vol% TiO₂/PEEK, 10 vol% TiO₂/5 vol% Al₂O₃/PEEK, and 20 vol% TiO₂/5 vol% Al₂O₃/PEEK. Hot pressing technique used to prepare samples; 50 MPa compression pressure at different compounding temperatures (370,380,390, and 400 °C).

II. Mechanical properties testing

Fracture strength was calculated from diametrical compression test [9] using Instron tensile machine with a crosshead speed of 5 mm min⁻¹. The following formula is used to calculate fracture strength [10]:

$$\sigma_f = \frac{2P}{\pi Dt} \quad (1)$$

Where

σ_f : Tensile fracture strength (MPa),

P: Crosshead load (N),

D: Specimen diameter (mm),

and t: Specimen thickness (mm).

microhardness values were calculated using microhardness tester (Digital Micro-Vickers Hardness tester TH714)for Beijing TIME High Technology Ltd./China).

III. In vivo inflammation test

In vivo tests used to describe the Biological Reactivity for prepared bio composite implants with highest mechanical properties estimated from mechanical testing (20 vol%TiO₂/5 vol%Al₂O₃/PEEK). Number and size of implants to be tested, have direct influence on the species of animal, which was chosen for a study. Some implant's designs are most commonly used in animal models like a screw type, or cylindrical (rod shaped).

The implants with cylindrical shape are dependent on exact fit in order to be stable within implantation zone at the bone to give accurate results regarding their effect on bone integration. [11]. In this work, cylindrical shape implants

were used. Four healthy local breed rabbits, aged (7-9) month old, weighted between 1–1.5 kg used in the current study. The rabbits were divided into two equal number groups:

In-group I, 5 mm bone segment was crafted at the mid shaft of radius bone and left without any further treatment, and considered as a control group.

In-group II, a same bone segment was crafted and filled with bio composite material which was prepared previously and considered as treated group.

All animals were injected with Acepromazine melete (10 mg/kg BW.) I/M as a tranquillizer. After a period of 10 minutes, every animal was injected again with a mixture of ketamine hydrochloride (35 mg/kg BW.), and xylazine (5mg/kg BW.) I/M. The local region of operation was surgically prepared, and the animal casted in a lateral recumbence, with a surgical incision of (3 cm) in length, which was made at the middle of the radius from the medial aspect. The subcutaneous tissue was cut; after that, a blunt dissection between the pronator teres muscle and flexor carpi radialis muscle was further made. The bone segment was cut by using an electrical saw, and then washed with normal saline. The segmental defect filled by composite sample in the treated group, and in control group the defect (fracture zone) left without additives.

All animals were subjected to radiography every 2 weeks and were euthanized after (2, 4, 6 and 8 weeks), and all specimens were analysed through histological path examination to observe the osteogenesis at the implant site.

IV. Radiological testing

Radiological test for all implants and control group have been carried out using Shimadzo digital x-ray machine with 500 MA/second and voltage of 100Kv and mas 4, grid 2016/ CRX10.

3. Results and Discussion

I. density and mechanical properties

Obtained density and mechanical properties are listed in Table 1, while figures from 1 to 3 shows relations between compounding temperature and density, fracture strength, and micro hardness respectively. These values is very close to natural bone properties [12-14], therefor no stress shielding effect should occur.

Table 1: comparison between measured properties and natural bone density and mechanical properties

Sp. composition	Compounding Temp. °C	Density (g/cm ³)	Fracture strength (MPa)	Microhardness kg/mm ²
cortical bone(compact)	-	1.6	131-224	33
cancellous bone(trabecular)	-	2.08	50–100	66
10 vol% TiO ₂ /PEEK	370	1.89	140	50
	380	1.894	150	51
	390	1.90	155	52
	400	1.92	162	54
20 vol% TiO ₂ /PEEK	370	1.93	170	56
	380	1.94	176	58
	390	1.946	178	59
400	370	1.95	180	60
	370	2.1	184	61
	380	2.14	188	61.5
10vol%TiO ₂ /5vol%Al ₂ O ₃ /PEEK	390	2.27	191	63
	400	2.31	192	63.7
	370	2.32	210	65
20vol%TiO ₂ /5 vol%Al ₂ O ₃ /PEEK	380	2.34	215	65.45
	390	2.36	218	66
	400	2.4	220	67

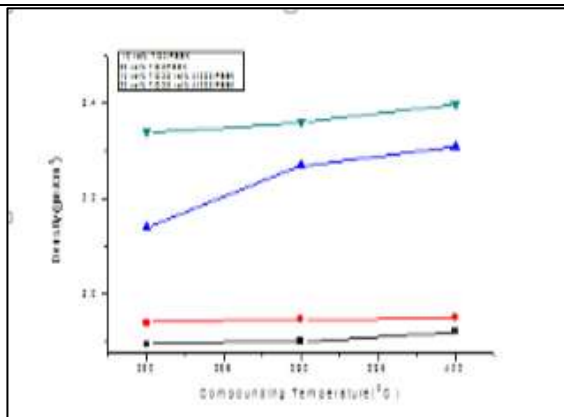


Figure 1: Relationship between density and compounding temperature for different compositions

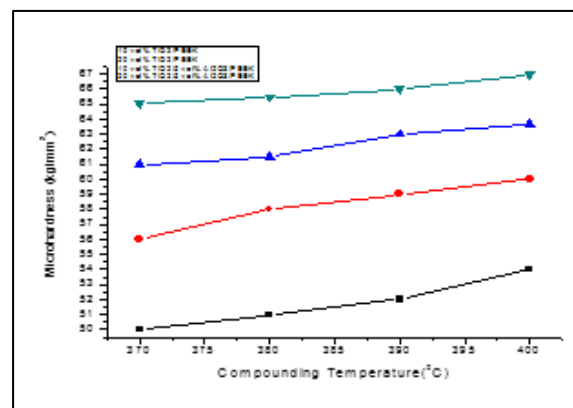


Figure 3: Relationship between microhardness and compounding temperature for different compositions

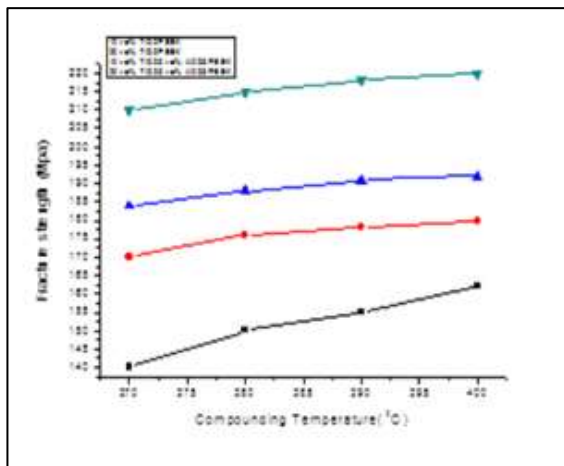


Figure 2: Relationship between fracture strength and compounding temperature for different compositions

I. Inflammations

Figures 4, 5, 6, and 7 are displaying a comparison between control group (fractured bone without implant) and those with implants. These figures show that no significant inflammation effect has resulted, with good healing outcome compared to control group.

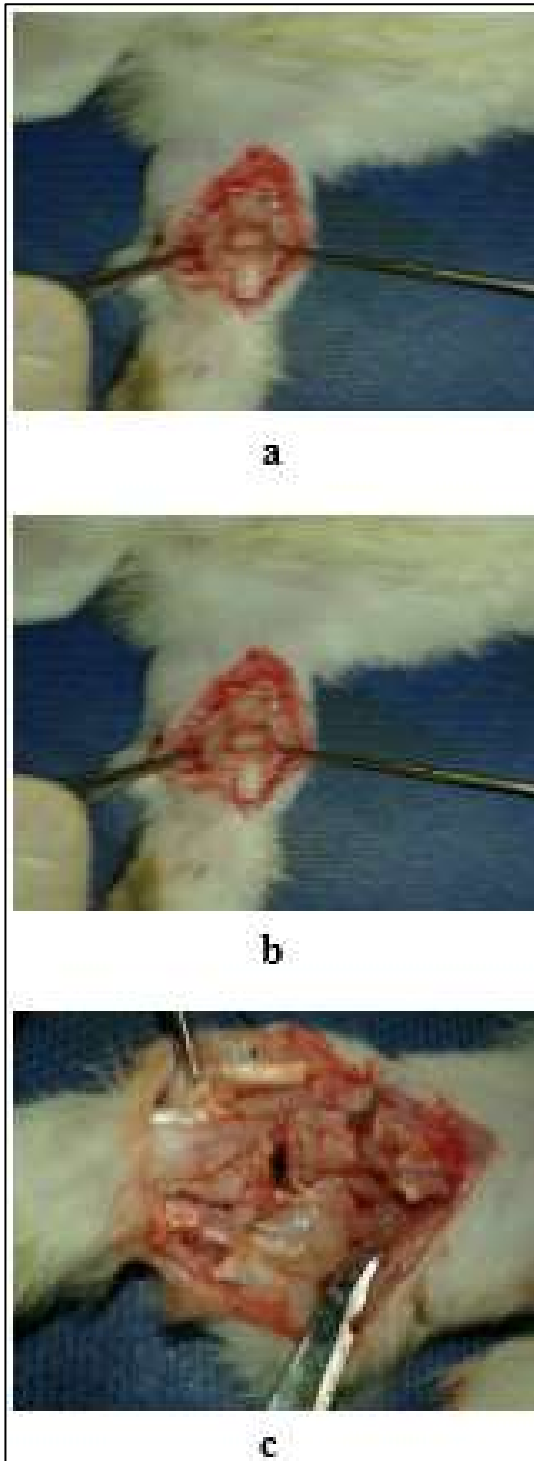


Figure 4: a, b: after 2 weeks implant, no inflammation occur, c: control group after 2weeks

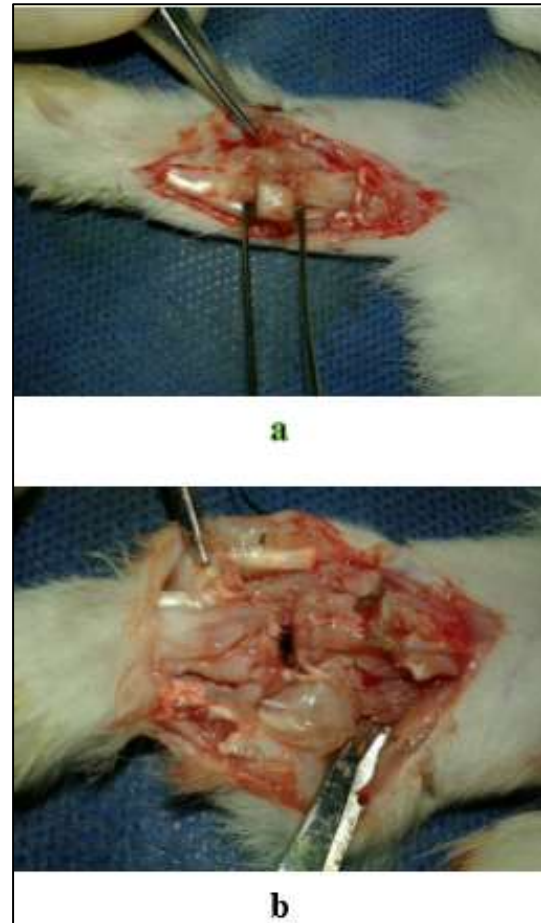


Figure 5: a: after 4 weeks implant, no inflammation occur, b: control group after 4weeks

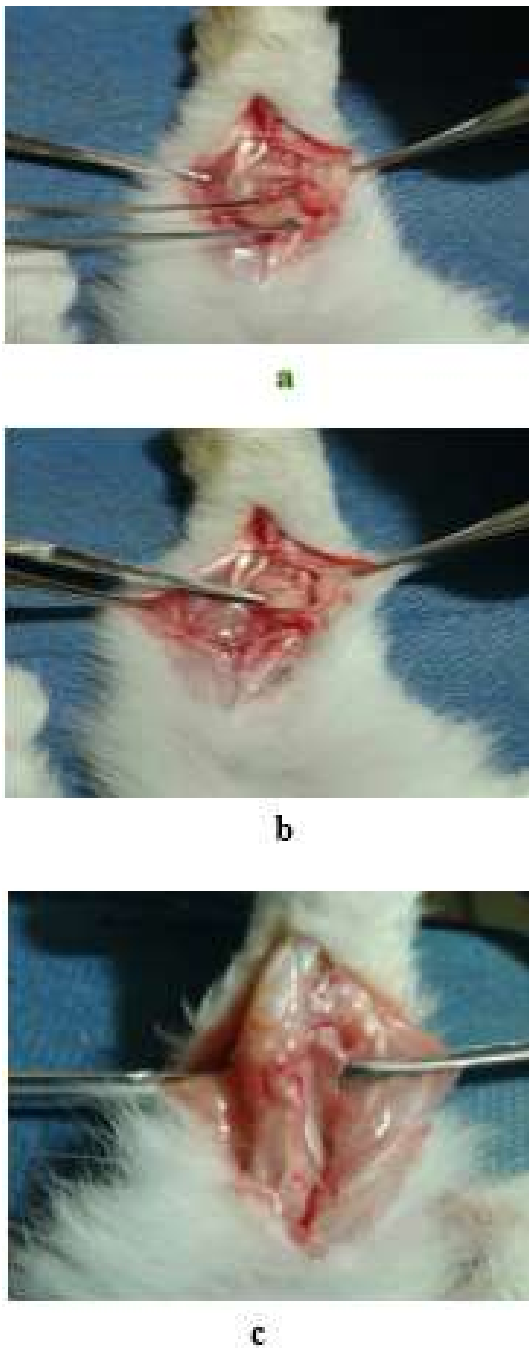


Figure 6: a,b: after 6 weeks implant, no inflammation occur, c: control group after 6 weeks

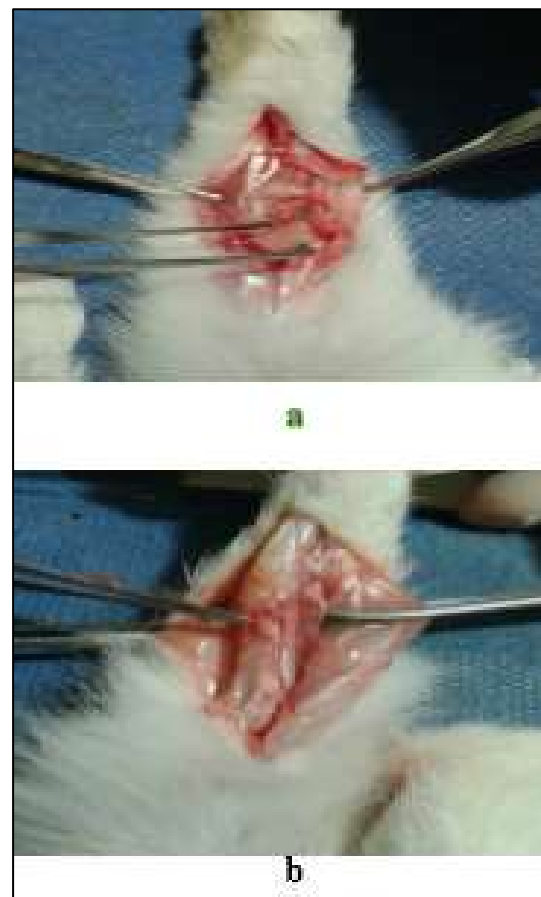


Figure 7: a- after 8 weeks implant, no inflammation occur, b- control group after 8 weeks

II. Radiological results

Figures 8, 9, 10, and 11 are displaying x-ray radiological results for both implant and control group at different implantation time (2,4,6,and 8 weeks). In Figure 8-a, the polymer implant occupies the gap between the cut ends (red arrow) immediately after operation, while in figure 8-b; 2 weeks after operation, slight reaction observed in control group (L) with periosteal reaction from ulna towards the bone segmental defect, and the polymer implant was still in its position (red arrow) (R).



Figure 8: x-ray results after 2 weeks implantation time, L: control group, R: treated group

In Figure 9; 4 weeks post operation, callus formation observed in control group (L) with periosteal reaction between radius and ulna; in treated group (R) the polymer implant stayed far from the gap (red arrow) and there was no callus formation with rounded proximal end. Radiological photo after 6 weeks presents in Figure 10; 6 weeks post operation, shows increase in periosteal reaction in control group (L) in attempt to bridging the bone defect, while in treated group (R) the cut end of the bone is rounded (non-union) and the polymer completely persists out of the bone defect. After 8 weeks implantation time, figure 1 presents x-ray results for implants and control group. 8 weeks post operation, increase callus formation and bridging the bone defect (not completely) in control group (L), while in treated group (R) the cut end of the bone is rounded (non-union) and the polymer completely persists out of the bone defect without any change in its radiological feature. All above results prove that these biocomposite have nontoxic or inflammation effect, which is one of the most important issues in biomaterials for bone repair and replacement.



Figure 9: x-ray results after 4weeks implantation time, a-L: control group, b-R: treated group



Figure 10: x-ray results after 6 weeks implantation time, a: control group, b: treated group



Figure 11: x-ray results after 8 weeks implantation time, a: control group, b: treated group

4. Conclusions

Estimated data shows that mechanical properties for investigated biocomposite are very close to natural bone mechanical properties, which is necessary to prevent stress shielding effect. On the other hand, implant interaction with animal model shows no inflammation effect, with better outcome than control group. Radiological test gave a good impression of injury regions. To support and confirm these we need more investigations with more implantation time (months), and histology study for tissue growth on biocomposite implant.

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