NIOBIUM OXIDE (Nb_2O_5) FILLED HIGH-MODULUS POLYETHYLENE EXTRUDABLE (HMPEX) COMPOSITES: TENSILE PROPERTIES

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Abstract

The present study reports the development of composites of high-modulus polyethylene extrudable (HMPEX) filled with niobium oxide (Nb_2O_5) for using in therapeutic applications. The effect of filler content on mechanical properties of composites processed with Nb_2O_5 contents in the range of 5-30 wt% was evaluated. Tensile strength tests were performed according to ASTM D 638. The tensile modulus values of composites increased significantly at high filler contents (15, 20, 25 and 30 wt%), where the modulus increased 18, 16, 14 and 24%, respectively, compared with the modulus of neat HMPEX. The yield strength showed slight improvement with the increase of filler content up to 20 wt%. At this concentration, it reached the maximum value, 10% higher than the value of HMPEX. The values of elongation at break and tensile strength decreased with the increase in filler content. All properties presented by the composites processed by extrusion were inferior to those of the composites prepared by compression molding. The neat HMPEX and the HMPEX composites processed with 5 wt% of Nb_2O_5 obtained by compression molding exhibited properties matching those required for medical grade UHMWPE resins, according to ASTM F648-00, making them potential candidates for medical applications.

Keywords: High modulus polyethylene extrudable, niobium pentoxide, biomaterial, therapeutic applications, tensile properties.

1. INTRODUCTION

Polymeric biomaterials, in the form of dental and medical implantable devices or implants, are widely used in the medical field for therapeutic applications. Examples of these applications are orthopedic implants, implantable cardiac defibrillators, coronary stents, and dental prosthetics or intraocular lenses [1 - 3]. Ultra-high-molecular-weight polyethylene (UHMWPE) is widely used to manufacture hip and knee implants for total joint arthroplasties [3 - 5]. Woven ribbons of

UHMWPE fibers are one the reinforcing materials most often used in dental composite resins for applications such as prosthodontics, restorations or periodontics [6 - 8]. In addition to its excellent biocompatibility, this polymer has an excellent combination of physical and mechanical properties, such as self-lubrication, low coefficient of friction, high impact resistance, high stress resistance, making it suitable for these applications [4, 5]. However, its processing is rather cumbersome compared with other grades of polyethylene. Generally, techniques based on the powder casting method are used. Usually, compression molding and ram extrusion are the techniques often used for consolidating forms [5, 9, 10], and gel spinning is used for obtaining fibers [11]. Thus, there is a need to develop less complex transformation processes, while maintaining the characteristic properties of this polymer [12 - 16].

The Petrobras Research & Development Center has developed a novel type of polyethylene, named high-modulus polyethylene extrudable (HMPEX) [17]. HMPEX has interesting characteristics, which make it a potential alternative material to UHMWPE for some applications. Its modulus is similar to the modulus of UHMWPE, but its processing properties are better than those presented by this polymer. Therefore, the production costs associated with HMPEX must be lower than those of UHMWPE.

Abrasion wear has a significant impact on the useful life of any implant. Wear debris accumulated at the interface of contact can accelerate the implant's mechanical failure. Those that migrate to peripheral tissues, when interacting with these tissue, can cause loosening of the implant, forcing its removal [18 -22]. Development of composites is one of the methods used to improve the wear resistance of polymeric materials [18]. Different type of fillers such as alumina [23], graphite, molybdenum disulfide, [24]; carbon nanofibers and nanotubes [25,26] have been added to biopolymers.

Niobium oxide, Nb_2O_5, is a mineral filler that is abundant in Brazil. It is commonly used in the preparation of dielectric ceramic materials, acoustic and electro-optic materials and optical glass, while in catalysis it is used as support phase or as promoter of chemical activity [27]. In recent years, many published reports describe its uses as biomaterial. Due to its properties of biocompatibility, high corrosion resistance, high mechanical strength and thermodynamic stability, it has been used as coating material for metallic biomaterials [28 - 30]. Nb_2O_5 filled titanium composites exhibit excellent biocompatibility and cell adhesion, and high mechanical strength, making it a promising material for use in orthopedic implants [31]. Besides being biocompatible, it is also a bioactive material. This property enables its use in the production of bioglass or ceramic glass for using as bone filler material or substitute material for bone tissues [30 - 34]. Some studies have shown the feasibility of using Nb_2O_5 as radiopacifying agent in the production of luting agents for endodontic treatment [35, 36]. Studies of Nb_2O_5 as a mineral filler in polymer composites for production of biomaterials are more recent. Leitune et al. [37] presented Nb_2O_5 as a novel filler for dental adhesive resin. These researchers developed Nb_2O_5 filled adhesive resins with improved tissue-resin bond strength compared with that of original resin. Young et al. [38] developed a Nb_2O_5-polydimethylsiloxane hybrid composite for application in coating on dental implants. Their results showed that tissue/implant interfaces could be optimized by adjusting the oxide content in the composite.

In this context, the aim of this study was to develop Nb_2O_5 filled HMPEX composites for using in biomedical applications. Since there is a relation between processing method, mechanical properties and wear resistance, the effect of filler content on mechanical properties of composites obtained by compression molding were compared with the properties obtained by extrusion.

2. EXPERIMENTAL

2.1 Materials

The matrix material used in this study was the high-modulus polyethylene extrudable (HMPEX) powder (Petrobras Research & Development Center, Rio de Janeiro, Brazil) (Melt Flow Index (21.60 kg, 190°C) = 0.84 g/10 min. Niobium pentoxide powder from Companhia Brasileira de Metalurgia e Mineração - CBMM) was used as filler. The α-tocopherol (E-vitamin) from Fagron was used as antioxidant.

2.2 Preparation of HMPEX/Nb_2O_5 composite samples by compression molding

Initially, the raw materials were dried in an oven with air circulation at 70ºC for 24 h. Then, different concentrations (5, 10, 15, 20, 25 or 30 wt%) of Nb_2O_5 were mixed with HMPEX by manual stirring, followed by ultrasonic agitation for 30 minutes. Finally, the mixtures were consolidated in the form of rectangular plates (109 mm x 107 mm x 4 mm), by using a press (Carver, 3851-OC) at 210ºC with force of 10 tons, warm-up time of 5 min, residence time of 7 min, and cooling at room temperature for 40 min.

2.3 Preparation of HMEPX/Nb_2O_5 composite samples by extrusion

Initially, HMPEX was mixed with 3 wt%, of vitamin E (VE) to prevent the polymer degradation by oxidation during processing. This mixture was introduced in a 500 ml flask and then an alcohol solution containing 72.4% absolute ethyl alcohol was added. The flask was immersed in a water bath maintained at 70°C with continuous stirring at 200 rpm, during 6 hours. After this period, the stirrer was turned off, and the temperature was maintained at 70°C until total evaporation of the residual alcohol solution. In the subsequent step, VE-doped HMEXP was pre-mixed with Nb_2O_5 (15 wt%) and, then, processed in a twin-screw extruder (Leistritz ZSE 18 MAXX) using a temperature profile, from feed to die of 180/190/200/210/220/230/240/250/260/270°C at 500 rpm screw speed and 2.0 kg/h feed rate.

2.4 Determination of tensile mechanical properties of the HMEPX/Nb_2O_5 composites

The tensile properties of neat HMPEX and reinforced HMPEX composites were determined according to ASTM D 638, using V-type test specimens, seven for each composition, obtained by machining with a milling machine (Roland, Desktop Engraver GX-350). The test was carried out in a Universal testing machine (Shimadzu, AGX-Plus 100 kN), at a crosshead speed of 50 mm/min with load of 5 kN.

3. RESULTS AND DISCUSSION OF RESULTS

Table 1 presents the results of tensile test of neat HMPEX (0% Nb_2O_5) and Nb_2O_5 filled HMPEX composites with different filler contents.

Table 1 shows that the elastic modulus and yield strength of composites increased with the increasing in filler content, while elongation at break and tensile strength decreased. This result matches the mechanical behavior generally observed in particle-filled polymeric composites [39 - 44]. The elastic modulus values of composites increased significantly at high filler contents (15, 20, 25 and 30 wt%) where the modulus increased 18, 16, 14 and 24%, respectively, compared with the neat HMPEX.

Table 1 - Tensile properties of HMPEX/ Nb_2O_5 composites obtained by compression molding.

Nb_2O_5 (wt%)	Elastic Modulus (MPa)	Yield Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)
0	1095.90±66.81	19.86±0.65	456.37±34.92	39.66±2.72
5	1063.22±33.95	19.89±1.12	371.81±37.33	31.08±3.28
10	1104.36±89.19	20.38±1.59	207.73±72.16	20.46±5.71
15	1293.92±47.76	21.09±0.17	139.67±88.08	18.85±2.30
20	1270.56±137.37	22.07±1.04	18.79±8.13	18.16±0.846
25	1247.99±80.98	19.34±1.09	34.99±3.99	6.63±2.76
30	1358.91±58.434	18.94±0.56	35.71±9.17	9.056±3.16

Figure 1 (a) illustrates the effect exerted by Nb_2O_5 content on the elastic modulus of each composite: for low filler content, the addition of Nb_2O_5 reduces the matrix cohesion, while for high contents, the reinforcing effect of the filler predominates [25]. The increment of elastic modulus value represents an increment in the stiffness of the composite [39]. Nb_2O_5 is a rigid filler, so it restricts the mobility of the polymer chains when inserted between them, so the stiffness increases [25, 42 - 46].

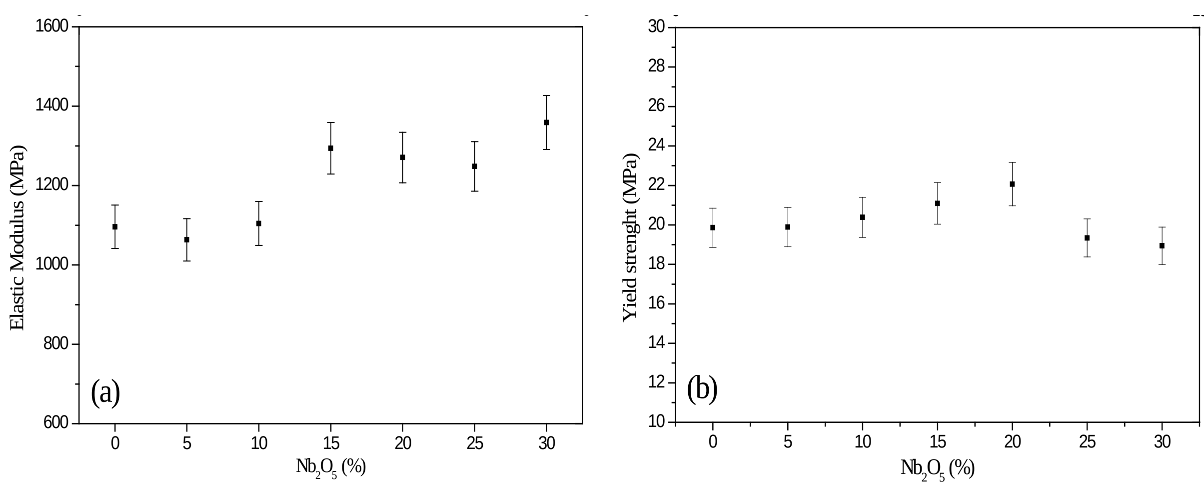

Figure 1 – The effect of filler content on the (a) elastic modulus, and (b) yield strength of HMEXP/Nb_2O_5 composites obtained by compression molding

Yield strength values of composites increased gradually, when the Nb_2O_5 content was increased, until a maximum value of 22.07 MPa for the composite filled with 20 wt % of Nb_2O_5 (Figure 1-b). This means an increase of 11% compared with that of neat HMPEX. For concentrations higher than 20 wt%, the yield strength values decrease gradually with the increase of Nb_2O_5 content. The minimum yield strength value reached 18.94 MPa, 4.6% below that of HMPEX. All composites, with the exception of that containing 30% of Nb_2O_5, exhibited values compatible with the minimum values of yield strength specified by ASTM F648-00 for medical grade UHMWPE resins (21 MPa for type 1 and 19 MPa for type 2).

The elongation at break of the composites decreased suddenly from 18% at 5 wt% of Nb_2O_5 to 96% at 20 wt% of Nb_2O_5 and remained practically constant above this filler content (Figure 2-a). Similar results were reported by Berçot [45] for niobium oxide filled polypropylene composites, and by Tavman [44] for aluminum-powder filled HDPE composites. The decreasing of elongation at break values is linked to the increment in stiffness promoted by the filler [39]. The HMPEX/5 wt% Nb_2O_5 and the neat UHMWPE present elongation at break values higher than the minimum required by ASTM F648-00 for medical grade UHMWPE resins (300%).

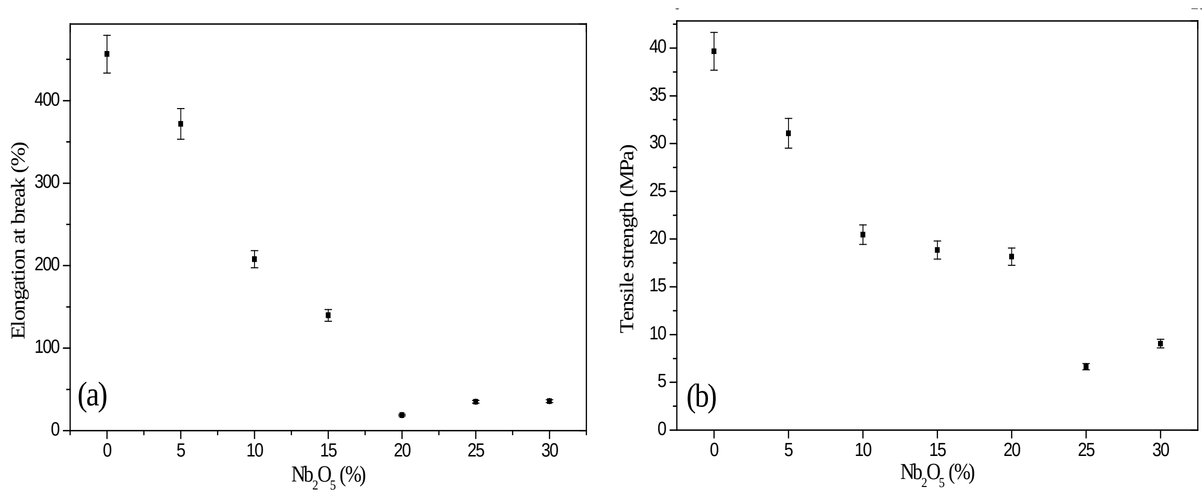

Figure 2 – The effect of filler content on the (a) elongation at break, and (b) tensile strength of HMEXP/ Nb_2O_5 composites.

The tensile strength decreased gradually with the increasing of filler content until 20 wt% content (Figure 2-b). However, there was an abrupt decrease at higher filler content. The maximum decrease, 83%, occurred at 25 wt% of Nb_2O_5 content. The reduction in tensile strength indicates the formation of filler particle agglomerates [40, 41]. This effect was stronger when a higher filler content (25 and 30 wt%) was introduced into the polymer. Formation of agglomerates, insertion of discontinuities with reduction of effective cross-sectional area of continuous phase and irregular distribution of particles can occur with increasing of the filler content. These effects account for the decrease in the tensile strength of composites [46]. The minimum tensile stress required for medical grade UHMWPE, for type 1 resin, is 35 MPa, and for type 2 is 27 MPa. According to these requirements, only neat UHMWPE and the composite processed with 5 wt% of Nb_2O_5 can be used for medical applications.

Table 2 shows the tensile properties of HMPEX/ 15% Nb_2O_5 composites prepared by the extrusion process (EP), compared to the values obtained for the composites prepared by compression molding (CM).

Table 2 shows that all tensile mechanical properties decreased in comparison to the corresponding values for composites obtained by compression molding. The standard deviation of elongation at break did not allow a reliable assessment of its value. This result suggests that there is a polymer degradation, when the composites are submitted to the extrusion process. This behavior should be investigated in depth.

Table 2 – Tensile properties of composites HMPEX/(15%)Nb_2O_5processed by compression molding (CM) and by extrusion process (EP)

Processing by	Elastic Modulus (MPa)	Yield Strength (MPa)	Elongation at Break (%)	Tensile Strength (MPa)
CM	1293.92 ± 47.76	21.09 ± 0.17	139.67±88.08	18.85±2.30
EP	839.31 ± 32.63	15.13 ± 1.12	474.20±364.22	11.96±8.61

4. CONCLUSIONS

Both neat HMPEX and the HMPEX/ 5% Nb2O5 exhibit properties suitable for medical grade UHMWPE resins according to ASTM F648-00.

The mechanical properties of HMPEX/ Nb2O5 composites prepared by compression molding are superior to those obtained by the extrusion process.

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