CAPÍTULO 6

APLICAÇÕES POTENCIAIS DE POLIETILENO EXTRUSÁVEL DE ALTO MÓDULO E SEUS COMPÓSITOS CARREGADOS COM ÓXIDO DE NIÓBIO

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RESUMO: O polietileno extrusável de alto módulo (HMPEX) da Petrobras apresenta propriedades físicas e mecânicas comparáveis às do polietileno de ultra-alto peso molecular (UHMWPE), contudo, é mais fácil de processar. O óxido de nióbio (Nb_2O_5) constitui uma carga mineral amplamente disponível no Brasil, sendo biocompatível e bioativo. No presente estúdio, avaliou-se o potencial de aplicação do HMPEX e seus compósitos carregados com Nb_2O_5. Procedeu-se à avaliação do efeito da incorporação de teores entre 5 e 20 wt% Nb_2O_5 nas propriedades de tração, térmicas e morfológicas dos compósitos de HMPEX moldados por compressão. Adicionalmente, procedeu-se à avaliação das propriedades de um compósito de HMPEX extrudado contendo 15 wt% Nb_2O_5, comparativamente ao seu homólogo moldado por compressão (MC). Resultados dos ensaios de tração evidenciaram que um incremento no teor da carga no MC promove aumento do módulo elástico e redução do alongamento na ruptura, da resistência à tração e da tenacidade. A resistência no escoamento apresentou baixa variabilidade. A análise termogravimétrica (TGA) e a calorimetria diferencial de varredura (DSC), não mostraram mudanças significantes na estabilidade térmica, ou nas propriedades térmicas dos compósitos. As micrografias obtidas por microscopia eletrônica de varredura (MEV) evidenciaram uma dispersão não homogénea da carga na matriz, e presença de aglomerados, cuja formação incrementa com o aumento

no teor de carga. A análise comparativa mostrou que o MC teve um melhor desempenho mecânico do que o composto extrudado (EX). Ao contrário do MC, o EX apresentou uma elevada diminuição da entalpia de fusão e do índice de cristalinidade. Apesar da presença de aglomerados, a microestrutura do EX exibe uma melhor dispersão e distribuição da carga no polímero do que a microestrutura do MC. Tanto o HMPEX como o MC com 5% de Nb_2O_5 cumprem os requisitos da norma ASTM 638F, demonstrando potencial para aplicações biomédicas. Por outro lado, o MC com 15% é adequado para aplicações que exijam um módulo elevado sem deformações significativas.
PALAVRAS CHAVE: polietileno de alto módulo extrusável, óxido de nióbio, biomaterial; material bioativo, propriedades de tração, carga mineral, propriedades térmicas...

POTENTIAL APPLICATIONS OF HIGH-MODULUS EXTRUDABLE POLYETHYLENE AND ITS COMPOSITES FILLED WITH NIOBIUM OXIDE

ABSTRACT: Petrobras' extrudable high-modulus polyethylene (HMPEX) has physical and mechanical properties comparable to those of ultra-high molecular weight polyethylene (UHMWPE), but is easier to process. Niobium oxide (Nb_2O_5) is a mineral filler that is biocompatible, bioactive, and widely available in Brazil. In this study, we evaluated the application potential of HMPEX and its composites loaded with Nb_2O_5. As part of this research, the effects of incorporating Nb_2O_5 on the tensile, thermal, and morphological properties of compression-molded HMPEX composites containing a filler of 5 to 20% by weight (wt%) were evaluated. In addition, the properties of an extruded HMPEX composite containing 15 wt% Nb_2O_5 were evaluated in comparison to its compression molded (CM) counterpart. Tensile testing revealed that increasing the filler content in the CMs led to an increase in elastic modulus and a decrease in elongation at break, tensile strength, and toughness, while yielding minimal variability in yield strength. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) revealed no significant changes in the thermal stability or thermal properties of the MCs. The micrographs obtained by scanning electron microscopy (SEM) showed the presence of agglomerates and an uneven filler distribution within the matrix. In terms of mechanical performance, the comparative analysis showed that the MC performed better than the EX. Unlike MC, the EX exhibited a significant reduction in melting enthalpy and crystallinity index. Agglomerated particles were also observed in the EX microstructure, but it presented superior filler dispersion and distribution in the polymer compared to the MC microstructure. On the other hand, MC containing 15% is suitable for applications requiring a high modulus without significant deformation.
KEYWORDS: High modulus extrudable polyethylene, niobium oxide; biomaterial; bioactive material, tensile properties, mineral filler, thermal properties.

INTRODUCTION

Ultra-high molecular weight polyethylene (UHMWPE) is an engineering material that has an optimal combination of physical and mechanical properties, including low coefficient of friction, self-lubrication ability, high impact resistance, high stress resistance and good chemical resistance. In addition, it exhibits excellent biocompatibility, making it a suitable

biomaterial for use in the production of medical implants (FARUK *et al.,* 2023; SHELLY, LEE & PARK, 2024; ZARIBAF, 2018; PATIL, NJUGUNA & KANDASUBRAMANIAN, 2020, HUSSAIN, AHMAD & SHEIKH, 2021).

UHMWPE is used to fabricate machinery components that require high resistance to abrasion, such as wear strips and scrapers, track rollers, abrasion protection strips, screw feeders, capping machine levers and energy-absorbing road safety systems, including plastic water-filler barriers, flexi traffic cones and anti-collision barriers (MLT ANALYTICS, 2024). Medical-grade UHMWPE features prominently in the manufacture of hip and knee implants for total arthroplasties, as well as shoulder and ankle replacement systems (BISTOLFI *et al*., 2021; PATIL, NJUGUNA, KANDSUBRAMANIAN, 2020; HUSSAIN, AHMAD & SHEIKH, 2021; BATTACHARYS, MUKHERJEE & PAL, 2020; SAWAE, 2018). In restorative dentistry, woven UHMWPE ribbons are among the most commonly used materials (AGRAWAL, 2014; DELIPERY, ALLEMAN & RUDO, 2017; SILVA, SILVA, CATUNDA, 2023).

The exceptionally high viscosity of UHMWPE poses a significant challenge to its processing using the conventional methods typically employed with other polyethylene grades, such as extrusion and injection molding. The most commonly used processing techniques for obtaining consolidated shapes from UHMWPE powder are compression molding and ram extrusion. The final forms are produced through machining (ZHANG & LIANG, 2018; WAHYUDI *et al*., 2018). The high pressures required in these processes lead to greater wear and tear of the equipment and higher production costs (YILMAZ, ELLINGHAM & TURNG, 2017). UHMWPE fibers are produced by gel spinning (LI & HU, 2018; XU *et al*., 2016). This technique requires meticulous and sometimes tedious operations for cleaning the equipment and recovering and recycling the solvent used to produce the fibers (YILMAZ, ELLINGHAM & TURNG, 2017).

Several studies have been conducted aiming to develop less complex transformation processes without compromising the characteristic properties of UHMWPE. Processing by microcellular injection molding in conjunction with supercritical plasticizers, such as nitrogen or carbon dioxide (YILMAZ, ELLINGHAM & TURNG, 2017) or processing in an eccentric rotor extruder at 180 °C while maintaining a continuous elongational flow and controlling residence time (CHEN *et. al*., 2021) are examples of this concept.

In this context, high-modulus extrudable polyethylene (HMPEX), a polyethylene grade developed by the Petrobras Research & Development Center, is a potential alternative to UHMWPE (ALVARES & HAAG, 2005; ALVARES *et al*., 1995). Since its modulus is comparable to that of UHMWPE and its processing properties are superior, the production costs associated with HMPEX are likely to be lower than those of UHMWPE. HMPEX fibers obtained through extrusion and spinning using conventional equipment demonstrate toughness values that make them well-suited for use in naval and offshore applications (ALVAREZ & HAAG, 2005).

The biomaterials utilized in implant fabrication, particularly joint replacement, must have a low wear rate. The progressive loss of material due to friction and wear has a significant impact on the useful life of an implant. The accumulation of debris at the implant-tissue interface can accelerate the mechanical failure of the implant and force its removal and replacement (TSUJIMOTO *et al*., 2018; BAENA, WU & PENG, 2015). Various fillers have been evaluated to improve the mechanical and tribological properties of these biomaterials. Examples are quasicrystaline metal powder based on aluminium alloys (FIGUEREIDO *et al*., 2022), graphenes and carbon nanotubes (APOSTU *et al*., 2023; PORTOLAS & KURTZ, 2014); solid lubricating materials such as molybdenum disulfide and polytetrafluoroethylene (PANIN *et al*., 2014); glass and carbon fibers (WANG & YIN, 2019); alumina nanofibers and alumina powder and (SENRA, MARQUES & SOUZA, 2020; HAJJAJ, 2012).. Addition of hydroxyapatite-based bioactive fillers has been demonstrated to stimulate enhanced bone growth and integration of implants with surrounding tissue (BALBINOT *et al*., 2020; MACUVELE, 2018). Niobium silicate particles have also been shown to promote the remineralization of dentin tissues without compromising the physicomechanical properties of tissue (NICO, MONTEIRO & GRAÇA, 2016). Bioactive fillers are used to improve implant-bone interaction after implantation.

Niobium pentoxide (Nb_2O_5), also called niobium oxide, is a mineral filler that is widely available in Brazil. It is used in diverse applications. Besides its role as a precursor to Nb metal and metal alloys, it has applications in diverse fields, including photocatalytic processing and the manufacture of capacitors, gas sensors, solar cells and photochromic devices (ASCENCIOS, 2021; CARVALHO *et al*.,2020; AUMAITRE *et al*., 2018). Niobium oxide exhibits elevated levels of mechanical resistance, corrosion resistance, and thermodynamic stability. Additionally, it is biocompatible and bioactive (SAFAVI *et al*., 2022). [36] These properties and characteristics make it an attractive material for use in the biomedical field. Research shows the potential use of this filler. A case in point is Nb_2O_5-filled titanium composites, which have been shown to have excellent biocompatibility and cell adhesion properties along with high mechanical strength (LI *et al*., 2016).

According to previous reports, the use of niobium oxide has shown promising results for the production of coating materials for dental implants and as a radiopacifying agent in cementation materials (SILVA *et al*., 2015; RAMÍREZ *et al*., 2011). Niobium oxide coatings on Ti6Al4V substrates can be used in dental, maxillofacial, and orthopedic implants (DINU *et al*., 2020). Dental adhesive resins based on Nb_2O_5 have demonstrated superior microhardness and radiopacity in comparison with commercial resins, along with stronger resin-tooth bonds (LEITUNE *et al*., 2013) Hybrid composites of Nb_2O_5-polydimethylsiloxane can provide an optimal soft tissue/implant interface, depending on the niobium oxide content (YOUNG *et al*., 2014). In light of the above considerations, the objective of this study was to evaluate the potential utilization of Nb_2O_5-filled HMPEX composites in biomedical applications. There is a well-known correlation between processing technique on the one hand and mechanical

properties and wear resistance on the other. Therefore, we also evaluated the mechanical properties of an extruded composite in comparison with those of a compression-molded composite.

EXPERIMENTAL

Figure 1 details the experimental procedure that was used in this study.

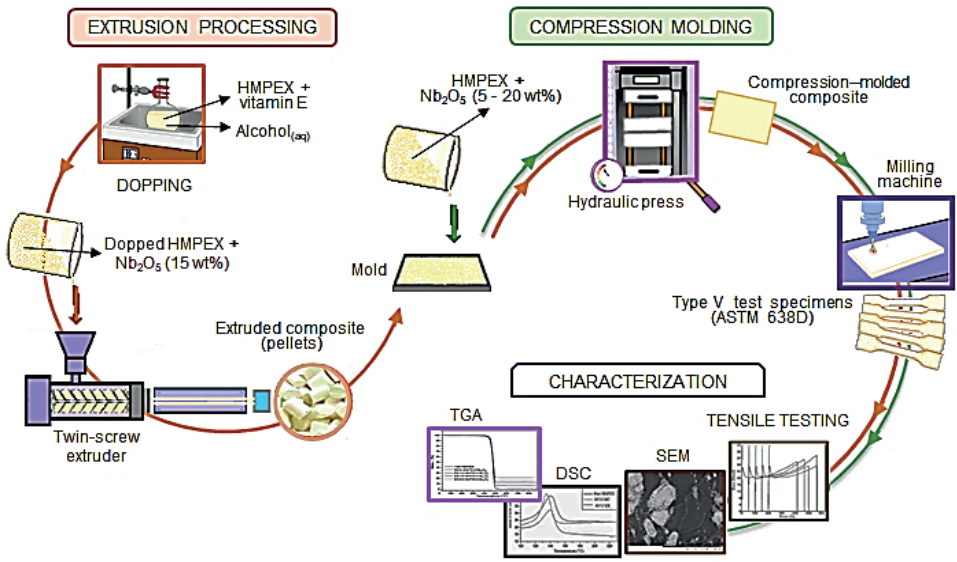

Figure 1. Experimental procedure used to development and characterization of HMPEX and its composites prepared in this study.

MATERIALS

In this study, high-modulus extrudable polyethylene (HMPEX) powder was provided by Petróleo Brasileiro S.A. (Petrobras). Its melt flow index (MFI) was 0.84 g/10 min at a temperature of 190 °C and a load of 21.60 kg. Calcinated niobium pentoxide (Nb_2O_5) powder, produced by Companhia Brasileira de Metalurgia e Mineração (CBMM, Brazil) was used as filler. This material has a wide particle size distribution, in the range of 0.2 to 100 μm, with 70% of particles larger than 1 μm. Vitamin E (α-tocopherol) powder supplied by Fagron was used as antioxidant in the preparation of the extruded composite.

PREPARATION OF HMPEX/Nb_2O_5 COMPOSITES BY COMPRESSION MOLDING

Initially, the raw materials were dried in a Marconi MA-035/1080/E oven under air circulation at 70 °C for 24 hours. The dried materials were subsequently passed through a 250 µm sieve to obtain materials with similar granulometry. This step aims to facilitate both the mixing of the materials and the subsequent homogenization of the resulting mixtures.

The sieving process was repeated until all the volume of material necessary for this study passed through the sieve. Before each repetition, the material was carefully macerated using a mortar and pestle.

Niobium oxide was added to the HMPEX at concentrations of 5, 10, 15 and 20% by mass (wt%). The mixtures were mechanically stirred for a few minutes to facilitate uniform dispersion. Subsequently, homogenization was conducted by ultrasonic agitation for 30 min in a Cristófoli ultrasound tank (42 kHz frequency) to ensure minimal porosity and to enhance the consolidation of the composite during the compression molding process.

Finally, rectangular plates measuring 109 mm x 107 mm x 4 mm of each composition were produced using a Carver/3851-OC hydraulic press at 210 °C with force of 10 tons. The hot compression molding execution program comprised: i) mold warm-up for 5 min at 210 °C, ii) residence time of 7 min, and iii) cooling time of 40 min at room temperature.

THERMAL PROPERTIES OF COMPRESSION-MOLDED HMPEX/Nb_2O_5 COMPOSITES

The termogravimetric analysis (TGA) and differential scanning calorimetry (DSC) methods were used to measure the thermal properties of composites. In order to evaluate the thermal stability of prepared materials and the residual mass that correspond to the actual filler content in each sample, TGA was used. To evaluate the effect of Nb_2O_5 content on the melting and crystallization behavior of HMPEX, the DSC was used.

TGA, derivative TGA (DTG) and DSC thermograms of pure HMPEX and its composites were recorded using a PerkinElmer STA 6000 simultaneous thermal analyzer, at a heating rate of 10 °C/min under a nitrogen atmosphere. The heating program consisted of a four-stage cycle: a first heating from 30 °C to 300 °C, followed by a 5-min isothermal hold at 300 °C, then cooling back to 30 °C, and finally a second heating (reheating) from 30 °C to 900 °C. The crystallinity index (was calculated by using the following equation:

$$X_c = \frac{\Delta H_m}{\left(\%p \times \Delta H_m^0\right)} \times 100 \qquad (1)$$

where, is the melting enthalpy of the sample (calculated from the integral of the endotherm peak in the DSC curve); ΔH_m^0 is the equilibrium melting enthalpy of polymer (melting peak of one gram of a 100% crystalline polymer); and $\%p$ is the weight percent of polymer in the composite.

MORPHOLOGICAL ANALYSIS OF HMPEX/Nb_2O_5 COMPOSITES

The morphological structure of HMPEX and composites was analyzed using a Hitachi TM3000 scanning electron microscope (SEM). This equipment operates with a Cu filament and acceleration voltages of 5 or 15 kV. SEM micrographs were obtained with 100X and 1000X magnification.

PREPARATION OF HMPEX/Nb_2O_5 COMPOSITE BY EXTRUSION PROCESSING

After evaluating the mechanical properties of the compression-molded composites, we chose the composite with 15 wt% niobium oxide (85/15 HMPEX/Nb_2O_5) to evaluate the effect of extrusion processing on these properties. First, a doping process was performed on the HMPEX using 3 wt% vitamin E (VE), to prevent oxidative degradation of the polymer during the extrusion process.

HMPEX and VE were mixed by mechanical stirring. The resulting mixture was transferred to a 500 ml volumetric balloon and a 72.4% ethanol solution was added. The mixture was maintained at a temperature of 70 °C in a water bath for 6 hours, with continuous stirring at 200 rpm. After this period, stirring was stopped and the temperature was maintained at 70 °C until complete evaporation of the residual alcohol solution.

The obtained VE-doped HMPEX was then mixed with Nb_2O_5 at a concentration of 15 wt%. The mixture, was subsequently processed in a Leistritz ZSE 18 MAXX twin-screw extruder. The extruder operated at a screw speed of 500 rpm, a feed rate of 2.0 kg/h, and a temperature profile (from the feed to the die) of 180/190/200/210/220/230/240/250/260/270 °C.

DETERMINATION OF TENSILE, THERMAL AND MORPHOLOGICAL PROPERTIES OF EXTRUDED HMPEX/Nb_2O_5 COMPOSITE

The tensile properties of the extruded composite (85/15 EX) were determined according to the procedure outlined in the subsection 2.3. Thermal characterization was conducted as detailed in subsection 2.4, and morphological analysis was performed in accordance with subsections 2.5.

RESULTS AND DISCUSSION

TENSILE PROPERTIES OF COMPRESSION-MOLDED COMPOSITES

Table 1 shows the results of the tensile tests of pure HMPEX (100 HMPEX) and the Nb_2O_5-filled HMPEX composites, which were prepared by compression molding. The composites are denoted as X/Y HMPEX/Nb_2O_5, where X indicates the HMPEX content and Y indicates the filler content, in percentage by weight (wt%). The tensile strength represents the maximum stress that the material can withstand before rupturing. The tensile strength values shown for the composites in the table are analogous to those of the tensile strength at break.

Sample[1]	Elastic modulus[3]	Yield strength[3]	Elongation at break[3]	Tensile strength[3]	Toughness[3]
	(MPa)	(MPa)	(%)	(MPa)	(MJ/m^3)
100 HMPEX[2]	922.4 ± 33.8	22.7 ± 0.7	455.8 ± 34.7	39.7 ± 2.7	63.1 ± 6.6
95/5 HMPEX/Nb_2O_5	917.9 ± 37.8	20.1 ± 1.1	380.0 ± 28.8	32.6 ± 2.7	47.8 ± 4.8
90/10 HMPEX/Nb_2O_5	955.0 ± 57.0	22.2 ± 1.3	6.1 ± 0.7	27.5 ± 1.7	23.3 ± 8.4
85/15 HMPEX/Nb_2O_5	1024.2 ± 8.4	21.1 ± 0.2	5.9 ± 0.2	28.7 ± 0,4	15.9 ± 9.7
80/20 HMPEX/Nb_2O_5	1057.0 ±72.6	22.1 ± 1.0	4.5 ± 0.6	26.9 ± 2.1	2.4 ± 1.0

[1]In X/Y HMPEX/Nb_2O_5, X is the percentage in weight (wt%) of HMPEX and Y is the wt% of Nb_2O_5; [2]100 HMPEX (pure compression-molded HMPEX) is the control sample; [3]Data are expressed as mean ± standard deviation

Table 1. Tensile properties of compression-molded HMPEX and HMPEX/Nb2O5 composites.

As shown in Table 1 and Figure 2(a), the elastic modulus of the composites exhibited a tendency to increase with rising filler content. This result is in agreement with the mechanical behavior of particle-filled polymeric composites, as documented in the literature (KHALAF, 2015; TASDEMIR & ERSOY, 2015; LARA-GONZÁLEZ et al., 2020).

The 85/15 and 80/20 HMPEX/Nb_2O_5 composites showed a significant increase in elastic modulus, with enhancements of 11.0% and 14.6%, respectively. The enhancement of the modulus with increasing filler was also observed with the addition of ZnO to the UHMWPE (CHANG *et al.*, 2011). An increase in the elastic modulus value indicates an increase in the rigidity of the composite (KHALAF, 2015;). [43] Nb_2O_5 is a rigid filler, so it restricts the mobility of the polymer chains when inserted between them, thus increasing the rigidity. (CHANG et al., 2011; PARVIN et al., 2013).

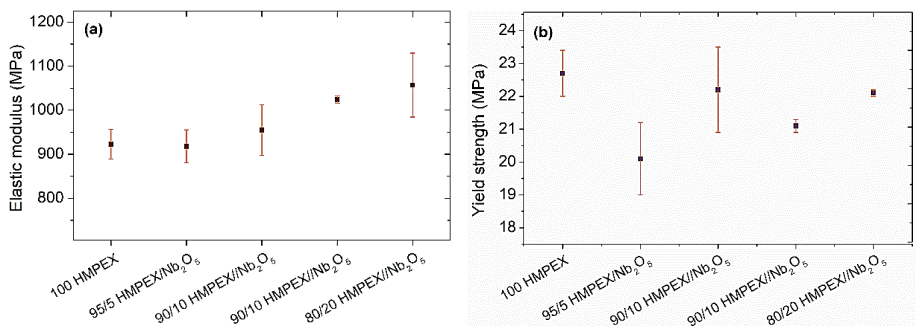

Figure 2. Effect of filler content on (a) modulus of elasticity and (b) yield strength of compression-molded HMPEX/Nb_2O_5. 100 HMPEX (pure compression-molded HMPEX) is the control sample. Data are expressed as mean ± standard deviate. In X/Y HMPEX/Nb_2O_5, X is the percentage by weight (wt%) of the HMPEXx; and Y is the wt% of the Nb_2O_5.

In contrast to the observed trend in the modulus, the yield strength did not demonstrate a correlation trend with the addition of the filler, as illustrated in Figure 2(b). The 90/10 and 80/20 HMPEX/Nb_2O_5 composites exhibited yield strength values close to those of pure HMPEX (100 HMPEX). The 95/5 and 85/15 HMPEX/Nb_2O_5 composites had yield strengths lower than that of pure HMPEX, exhibiting reductions of 10% and 7%, respectively. These results can be attributed to the poor interfacial adhesion between the filler and matrix of these composites. However, all composites exhibited values consistent with the yield strength values specified for medical grade UHMWPE resins according to ASTM F648: 21 MPa for grade type 1 and 19 MPa for grade type 2.

As shown in Figure 3(a), the tensile strength gradually decreased with increasing filler content, up to 31% at 10 wt% Nb_2O_5 (90/10 HMPEX/Nb_2O_5). For higher niobium oxide contents, the values tended to remain constant. The reduction in the tensile strength indicates the formation of filler particle agglomerates and low interphase adhesion (TASDEMIR & ERSOY, 2015; PARVIN *et al.*, 2013; SALAMA *et al.*, 2022). The formation of agglomerates, insertion of discontinuities with a reduction in the effective cross-sectional area of the continuous phase, and irregular distribution of particles can occur with increasing filler content. These effects accounted for the decrease in the tensile strength of the composites (CHANG *et al.*, 2011). The minimum tensile stress required for medical grade UHMWPE for type 1 resin is 35 MPa, and that for type 2 is 27 MPa. According to these requirements, only pure HMPEX and the composite with 5% Nb_2O_5 (90/5 HMPEX/Nb_2O_5) can be used for biomedical applications.

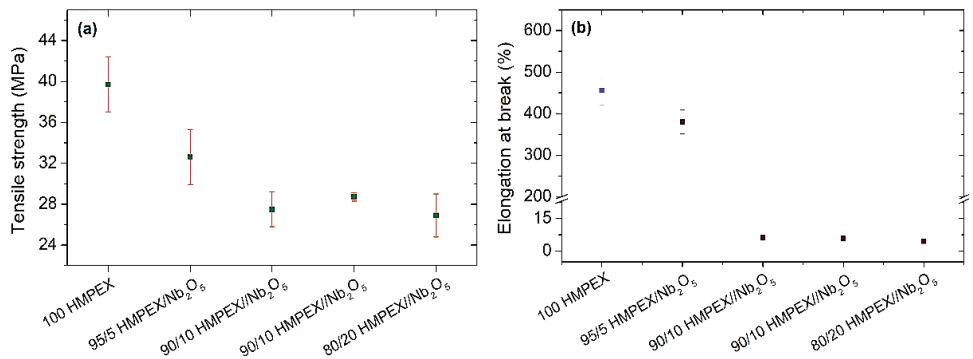

Figure 3. Effect of filler content on the (a) elongation at break, and (b) tensile strength of compression-molded HMEXP/Nb_2O_5 composites. 100 HMPEX (pure compression-molded HMPEX) is the control sample. In X/Y HMPEX/Nb_2O_5, X is the wt % of the matrix and Y is wt % of the filler. The bars represent the standard deviation of the mean.

Figure 3(b) shows that the elongation at break of the composites experienced a sudden decrease from 18% at 5 wt% Nb_2O_5 to 96% at 20 wt% Nb_2O_5 (95/5 and 80/20 HMPEX/Nb_2O_5, respectively). Similar results were reported for aluminum powder-filled HDPE composites (TAYMAN, 1996), carbon black-filled HDPE/propylene-ethylene-random-

copolymer composites (LI *et al.*, 2023), and graphene oxide (GO)-filled UHMWPE composite (CHEN et al. 2012).

The decrease in elongation at break values is related to an increase in the stiffness promoted by the filler (Khalaf, 2015). [43] Despite this, the 95/5 HMPEX/Nb_2O_5 and pure HMPEX (100 HMPEX) exhibited elongation at break values higher than the minimum value required by ASTM F648-00 for medical grade UHMWPE resins, which is 300%.

Figure 4 shows a linear pattern of the loss of the material's ability to absorb energy with rising niobium oxide concentration. The reductions in toughness were 25%, 64%, 75% and 96% at 5, 10, 15, and 20 wt% Nb_2O_5, respectively. This result was expected, because inorganic fillers generally produce harder and more brittle materials when incorporated in polymeric matrices (Zuiderduin, *et al.,* 2003; BERÇOT, 2018). Chen *et al.* (2012) also observed reduced toughness of UHMWPE when graphene oxide was adding to the biopolymer.

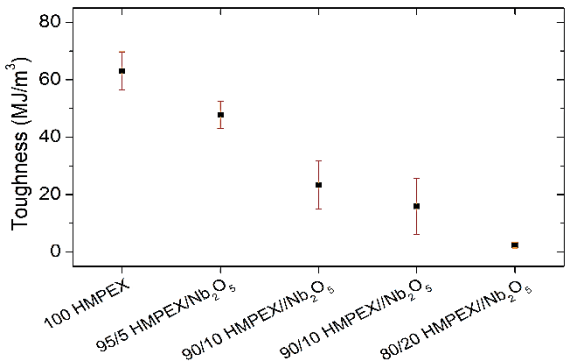

Figure 4. Effect of filler content on the toughness of compression-molded HMPEX/ Nb_2O_5. 100 HMPEX (pure compression-molded HMPEX): control sample. The bars represent the standard deviation of the mean.

As indicated by the results previously presented, the tensile tests of HMPEX/Nb_2O_5 composites demonstrated a general tendency towards a reduction in tensile properties with the rising incorporation of fillers, with the exception of the elastic modulus, which tended to increase at 10 wt% filler content. The findings of this study are consistent with data reported in other studies (LARA-GONZÁLEZ, 2020; KHALAF, 2015; PARVIN *et al.*, 2013; CHANG *et al.*, 2011).

We also observed that the tensile properties of both the pure HMPEX and the composite with a 5 wt% Nb_2O_5 content satisfied the requirements of ASTM F648, the standard for medical grade UHMWPE powder.

THERMOGRAVIMETRY ANALYSIS (TGA) AND DIFFERENTIAL SCANNING CALORIMETRY (DSC) OF COMPRESSION-MOLDED HMPEX/NB_2O_5 COMPOSITES

The results on thermal stability of the HMPEX/Nb_2O_5 composites with different filler contents are presented in Table 2,

Sample[1]	T_{anset}	T_{90} [4]	T_{50} [5]	T_{max} [6]	Residual mass
	(ºC)	(°C)	(°C)	(°C)	(wt%)
100 HMPEX[2]	403	456	477	481	0.0
95/5 HMPEX/Nb_2O_5	416	452	476	477	6.3
90/10 HMPEX/Nb_2O_5	403	463	475	478	11,9
85/15 HMPEX/Nb_2O_5	397	453	477	480	15.3
80/20 HMPEX/Nb_2O_5	417	456	476	478	21.1

[1]In X/Y HMPEX/Nb_2O_5, X is the wt% of HMPEX and Y is the wt% of Nb_2O_5; [2]100 HMPEX (pure compression-molded HMPEX) is the control sample; [3]degradation onset temperature, [4,5]temperatures at 90% and 50% of mass loss, respectively; [6]temperature at the maximum rate of degradation.

Table 2. Thermal stability of compression-molded MPEX/Nb_2O_5 composites.

The table lists the T_{anset}, T_{90} and T_{50} of the samples. These parameters correspond to the onset degradation temperature and temperatures at a mass loss of 90% and 50%, respectively.

As shown in Table 2, the values of T_{anset}, T_{90}, T_{50} and T_{max}, present little variation. This indicates that incorporating Nb_2O_5 into HMPEX did not significantly alter the material's thermal stability. However, in the case of a polypropylene matrix, adding niobium pentoxide was reported to slightly increase thermal stability (LACERDA, 2019).

The residual mass values in Table 6, indicate that the actual filler content of the HMPEX/Nb_2O_5 composites closely aligns with the formulated filler content. This suggests that the material preparation and mixing methods described in Section 2.2 help reduce losses and variations in the composition of the composite constituent materials.

As showed in Table 3, the increase in the niobium oxide (Nb_2O_5) content in the HMPEX matrix did not result in substantial alterations to the melting and crystallization temperatures, T_f and T_c, respectively. In a similar manner, the degree of crystallinity, X_c, and the melting enthalpy, ΔH_f exhibited no significant variations in their values as the Nb_2O_5 content increased.

Sample[1]	T_f [3]	ΔH_f [4]	T_c [5]	X_c [6]
	(ºC)	(J/g)	(°C)	(%)
100 HMPEX	141	563	105	76,00
95/5 HMPEX/Nb_2O_5	142	581	102	64,83
90/10 HMPEX/Nb_2O_5	138	574	105	60,74
85/15 HMPEX/Nb_2O_5	140	463	105	70,72
80/20 HMPEX/Nb_2O_5	140	494	106	70,69

[1]In X/Y HMPEX/Nb_2O_5, X is the wt% of HMPEX and Y is the wt% of Nb_2O_5; [2]100HMPEX (pure compression-molded HMPEX) is the control sample; [3]melting temperature; [4]melting enthalpy; [5]cristalization temperature; [6]index crystallinity.

Table 3. DSC data of compression-molded HMPEX/Nb_2O_5 composites.

MORPHOLOGIC ANALYSIS BY SCANNING ELECTRON MICROSCOPY (SEM) OF COMPRESSION MOLDED COMPOSITES

Figures 5 (a-d) show the fracture micrographs at 100x and 1000x magnification of compression molded HMPEX/ Nb_2O_5 composites samples containing 5 wt%, 10 wt%, 15 wt%, and 20 wt% Nb_2O_5.

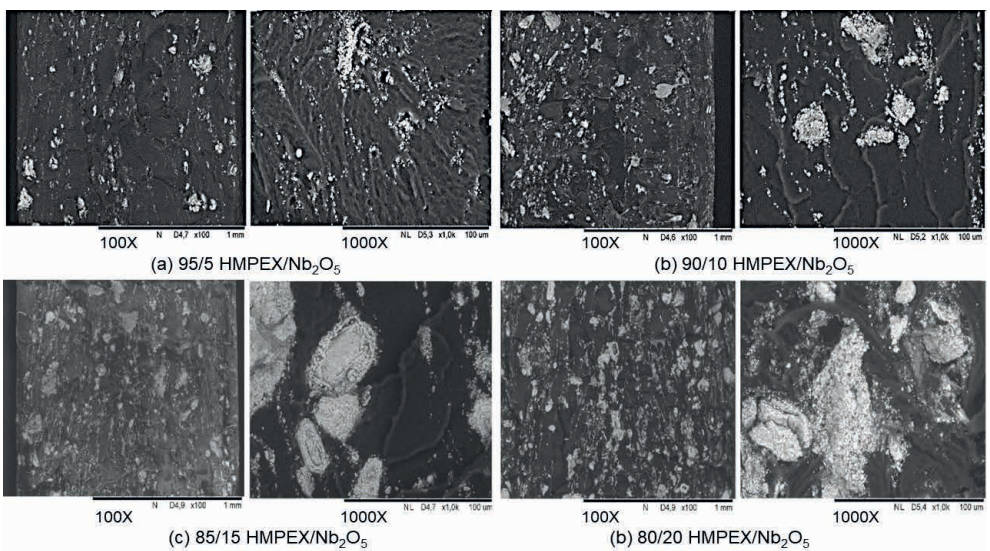

Figure 5. SEM micrographs of HMPEX/Nb_2O_5 composite samples with (a) 5 wt% (95/5 HMPEX/Nb_2O_5); (b) 10 wt% (90/10 HMPEX/Nb_2O_5); 15 wt% (85/15 HMPEX/Nb_2O_5); and (d) 20 wt% (80/20 HMPEX/ Nb_2O_5) filler content. Each sample is presented with two micrographs: one at 100X magnification (on the left) and the other at 1000X magnification (on the right).

A thorough examination of the micrograph reveals that, in general, compression molding did not yield composites with optimal dispersion and distribution of the inorganic phase within the HMPEX matrix. The presence of aggregates is indicative of inadequate dispersion of filler particles. Furthermore, it was noted that the distribution of filler particles was non-uniform.

COMPARATIVE ANALYSIS OF TENSILE, THERMAL AND MORPHOLOGICAL PROPERTIES OF 85/15 HMPEX/NB_2O_5 COMPOSITES

This section presents a comparative analysis of the tensile, thermal, and morphological properties of 85/15 HMPEX/Nb_2O_5 composite prepared using either compression molding or extrusion.

The findings of the tensile tests of the 85/15 HMPEX/Nb_2O_5 composite containing obtained by extrusion (85/15 EX), are presented in Table 4. For the purpose of comparison, the tensile properties of composite 85/15 HMPEX/Nb_2O_5 produced by compression molding (hereafter referred to as 85/15 CM) and of pure HMPEX (100 HMPEX) are also displayed. To facilitate a comparative analysis of the tensile properties of these materials, a graphical representation of the result kit's findings is presented in Figure 4.

Sample	Elastic modulus[1]	Yield strength[1]	Tensile strength[1]	Elongation at break[1]	Toughness[1]
	(MPa)	(MPa)	(MPa)	(%)	(MJ/m^3)
100 HMPEX[2]	922.4 ± 33.8	22.7 ± 0.7	39.7 ± 2.7	455.8 ± 34.7	63.1 ± 6.6
85/15 CM	1024.2 ± 8.4	21.1 ± 0.2	28.7 ± 0.4	5.9 ± 0.2	15.9 ± 9.7
85/15 EX	776.0 ± 17.2	15.1 ± 0.2	24.8 ± 1.4	8.2 ± 0.8	45.1 ± 38.2

[1]Data are expressed as mean ± standard deviation.[2]100 HMPEX (pure compression-molded HMPEX) is the control sample

Table 4. Tensile properties of composites with 15 wt% Nb_2O_5 content obtained by by extrusion (85/15 EX) comparing with the compression molded composite (85/15 CM).

As shown in Table 2 and Figures 4 (a), (b) and (c), the elastic modulus, yield strength and tensile strength, respectively, of the extruded composite (85/15 EX) were lower than those of the compression-molded composite (85/15 CM). The 85/15 EX exhibited a 16% decrease in elastic modulus, while the 85/15 CM exhibited an 11% increase.

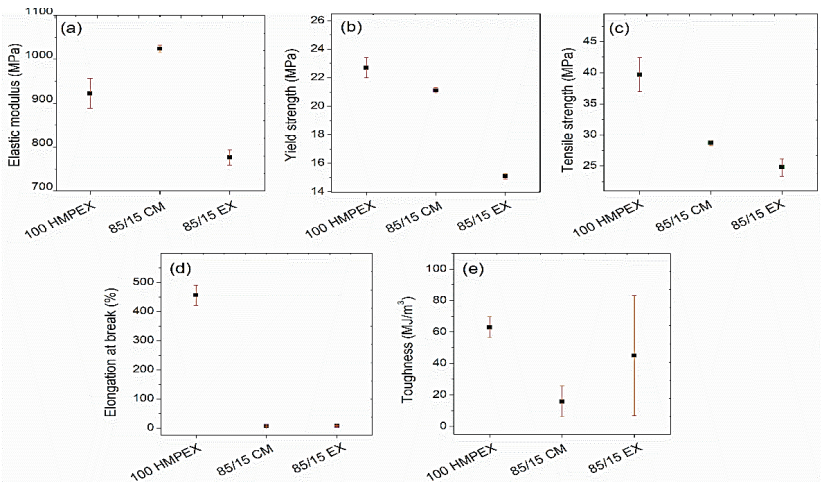

Figure 6. Comparison of tensile properties of the 15 wt% Nb_2O_5– filled- HMPEX composites obtained by extrusion processing (85/15 EX) or by compression molding (85/15 CM). (a) elastic modulus, (b) yield strength, (c) tensile strength, (d) elongation at break, and (e) toughness. 100 HMPEX (pure compression-molded HMPEX) is the control sample. Bars represent the standard deviation of the mean.

Both composites, 85/15 CM and 85/15 EX, underwent a decrease in yield strength. However, 85/15 EX exhibited a 24% decrease, while 85/15 EX exhibited a 7% decrease. This was also the case for tensile strength; 85/15 EX experienced a 38% decrease, which is much greater than the 10% decrease observed in the 85/15 CM. The elongation at break of both composites showed an abrupt reduction, with the 85/15 EX exhibiting elongation at break percentages greater than the 85/15 CM, of 8.2% and 5.9%, respectively, in accordance with Table 4 and Figure 6(d).

It is not feasible to assert that the absorption capacity of 85/15 EX increased in comparison with 85/15 CM, since the standard deviation associated with toughness is too high (refer to Table 4 and Figure 6(e)). This significant experimental error suggests that the conditions employed did not produce a homogeneous material.]

This comparative analysis of the composites revealed that the extruded composite exhibited inferior mechanical performance than the compression-molded composite. This result can be attributed to the presence of poor interfacial interactions between the filler and matrix, as well as the uneven distribution of the filler in the extruded composite. The reduction in tensile properties suggests it is necessary to investigate other processing methods for the composites.

The TGA results for the 85/15 HMPEX/Nb_2O_5 composite obtained through the compression molding (CM) and extrusion (EX) processes are presented in Table 5.

Sample[1]	T_{onset} [3]	T_{90} [4]	T_{50} [5]	T_{max} [6]	**Residual Mass**
	(°C)	(°C)	(°C)	(°C)	(wt%)
100 HMPEX[2]	403	456	477	481	0.0
85/15 CM	397	453	477	480	15.32
85/15 EX	401	453	475	477	15.81

[1]In X/Y HMPEX/Nb_2O_5, X is the wt% of HMPEX and Y is the wt% of Nb_2O_5; [2]100 HMPEX (pure compression-molded HMPEX) is the control sample; [3]degradation onset temperature, [4,5]temperatures at 90% and 50% of mass loss, respectively; [6]temperature at the maximum rate of degradation.

Table 5. Thermal stability of 85/15 HMPEX/Nb_2O_5 composites prepared by compression-molding (85/15 CM) and prepared by extrusion process (85/15 EX)

As illustrated in the table, the temperatures T_{onset}, T_{90}, T_{50}, T_{max} and of 85/15 HMPEX/Nb_2O_5 composites not exhibit significant variations regardless of the production method. Both 85/15 HMPEX/Nb_2O_5 composites, compression-molded (85/15 CM) and extruded (85/15 EX), demonstrated load content that closely matched their original formulated content.

The results of the DSC analysis of the 85/15 HMPEX/Nb2O5 composites obtained by compression molding (85/15 CM) and extrusion (85/15 EX) are presented in Table 6. This table includes the T_f, ΔH_f, T_c, and T_c values of the composites and the pure compression-molded HMPEX.

Sample[1]	T_f [3]	ΔH_f [4]	T_c [5]	X_c [6]
	(ºC)	(J/g)	(°C)	(%)
100 HMPEX	141	563	105	76,00
85/15 CM	140	463	105	70,72
85/15 EX	135	434	111	58,82

[1]In X/Y HMPEX/Nb_2O_5, X is the wt% of HMPEX and Y is the wt% of Nb_2O_5; [2]100HMPEX (pure compression-molded HMPEX) is the control sample; [3]melting temperature; [4]melting enthalpy; [5]cristalization temperature; [6]index crystallinity.

Table 6. DSC data of 85/15 HMPEX/Nb_2O_5 composites prepared by compression-molding (85/15 CM) and prepared by extrusion process (85/15 EX).

As shown in Table 6, the extruded 85/15 HMPEX/Nb2O5 composite (85/15 EX) exhibits variations in its melting and crystallization temperatures (T_f and T_c, respectively) when compared to the compression-molded composite (85/15 CM) and pure HMPEX. It is evident that the ΔH_f and X_c values decreased when the composite was extruded compared to the compression molded composite.

Figure 7 illustrates the SEM micrograph of the fracture surface of a sample of the 85/15 HMPEX/Nb_2O_5 composite, which was generated through extrusion.

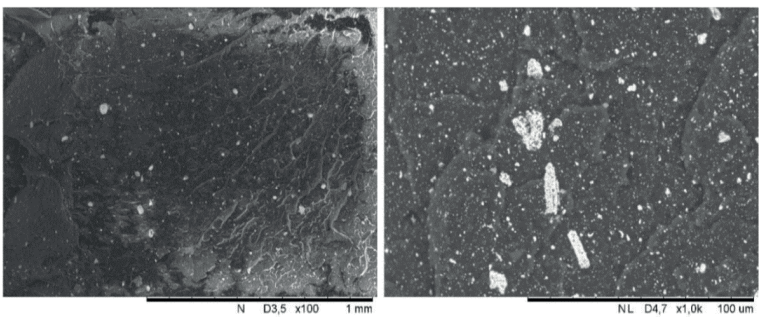

Figure 7. SEM micrographs of 85/15 HMPEX/Nb_2O_5 composite sample produce by extrusion processing (85/15 EX). The image on the left is magnified 100 times, while the image on the right is magnified 1,000 times.

Compared to the SEM micrographs of compression-molded 85/15 HMPEX/Nb_2O_5 composite presented in Figure 5 (c), the micrograph in Figure 7 shows that the extruded 85/15 HMPEX/Nb2O5 composite had a better distribution and dispersion of niobium pentoxide in the polymer matrix. However, it is still possible to observe the formation of some niobium pentoxide agglomerates.

CONCLUSIONS

The results of tensile test indicate that neat HMPEX and the HMPEX composite filled with 5 wt% niobium oxide content are potential candidates for biomedical applications. HMPEX composites processed with 15 or 20wt% niobium oxide content can be used in applications requiring high modulus and low deformation.

Comparative analysis of the tensile properties revealed that with the exception of elongation at break, the compression-molded composites exhibited superior mechanical performance. The thermal stability and thermal properties of the composites is not affected by niobium oxide content. It is evident that both processing types result in a reduction of melting enthalpy and crystallinity index. However, extrusion results in a higher level of reduction of these parameters. The presence of aggregates is observed in the microstructure of both compression-molded and extruded composites. Extruded composite demonstrates superior dispersion and distribution of filler in the HMPEX matrix, exhibiting lower size and number aggregates compared to compression-molded composite...

AUTHOR'S CONTRIBUTION

The contributions of each author to this manuscript are described below:

João Pedro Coré Pinto: Development of the experimental part related to the development and characterization of the composites. **Nancy Isabel Alvarez Acevedo**: Analysis and interpretation of the data; writing and editing of the manuscript. **Marisa Cristina Guimarães Rocha**: Conceptualization and design of the project; analysis of the results obtained; revision of the manuscript. **Noemi Tatizawa**: Synthesis of high-modulus extrudable polyethylene (HMPEX).

The final version of the manuscript was approved by all authors.

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DISCLOSURE STATEMENT

The authors report there are no competing interests to declare.

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