Development and surface properties of polyamide 6 and quasicrystal composite coatings recycled via spin coating technique

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Abstract
In search to meet technological demands, material studies as well as techniques to process them have been increasing worldwide, thus seeking to unite lower processing costs, the best properties of the materials involved, reuse of resources and more sustainable means. Thus, the development of new materials, coatings and composites have stood out in the market. For this work, we fetch the development and characterization of polyamide 6 composite coatings with quasicrystalline AlCuFe fillers, recycled through the spin coating technique, since that little is known about the interaction between these two materials, which have excellent isolating properties, and little is observed about the use of this technique to produce coatings with metallic loads. The recycled AlCuFe quasicrystalline alloy was manufactured by conventional induction casting. Polyamide 6 coatings with quasicrystalline particles were developed with additions of 0, 1, 3 and 5 (vol.%) quasicrystal, via Spin Coating technique. Thermogravimetric analyses (TG), differential scanning calorimetry (CSD), average roughness (RA), contact angle, surface energy and scanning electron microscopy (SEM) were performed to characterize this material. Being observed an increase in the thermal stability of the composite, reduction in average roughness (Ra) and surface energy values, with increase in quasicrystal fraction in the polymer matrix. In addition to hydrophobicity and oleophilic character of the samples with the addition of quasicrystalline loads. It can be concluded, in general, that the Spin Coating technique is effective in the production of polyamide composite coatings 6 with recycled quasicrystals, presenting interesting surface properties with potential for further studies in innovative applications, such as semiconductors and surface coatings.

1. Introduction
Surface properties, surface chemistry, techniques that modify surface properties to expand the applications and technologies of materials have been widely studied, properties such as wettability, roughness and surface energy became fundamental to the understanding of the interaction between materials and possible applications [1, 2]. Therewith, the promising combinations of polymer matrix composites with loads, either metallic, ceramic and currently quasicrystalline, to stimulate better surface properties, as well as low density, high wear resistance, among other properties, gain prominence, along with the best techniques to process them. Therefore, a challenge for science and engineering is the development of new materials that accompany technological advances and with lower cost-benefit [3–5].
Among the most used polymers can be mentioned polyamides or nylon, whose consumption in the production of parts in Brazil is 24,000 tons year$^{-1}$ [6]. It has excellent thermal resistance characteristics, good chemical resistance, high wear resistance, low density, and reduced cost, making it an excellent material for various applications, from films and gears to engine housings, presenting multifunctionality in the industrial sector [7, 8]. In addition, studies on optical and electrical properties of Polyamides have been increasing, such as semiconductor polymers, organic film transistors and polymer matrix composites with semiconducting properties [9, 10]. Research shows that the change in the crystallinity of these polymeric materials, with the addition of charges, can directly influence their semiconductor properties [9, 11, 12].

The most common loads used to modify the crystallinity and consequently the semiconductor properties of the materials are oxides, nitrates and carbides, such as TiO2, ZnO, BN, that is, all ceramic materials that require more processing energy and present hydrophilic properties [11, 13].

Little is known about the interaction and modification of the crystallinity of polymeric materials using recycled quasicrystalline fillers, in order to change their semiconductor properties, however, it is known that there is a lower energy consumption and lower gas emission in the recycling process of the materials used in the alloy, encouraging sustainability and without altering the alloy properties. Furthermore, these quasicrystalline alloys, when used as fillers in polymeric matrices, reduce the hydrophilic percentage of the polymers, a behavior that does not happen with fillers of ceramic materials [14, 15].

A load that has been studied for more than two decades is quasicrystalline alloys (QC) which have characteristics such as low surface energy, low coefficient of friction, high resistance to wear, corrosion, and high hardness. The combination of these properties and the fact that it cannot be used alone, due to its fragility, favors its application as a load in composite materials and coatings [16–20]. Thus, several studies have demonstrated the feasibility of using QC loads for the manufacture of composites with polymeric matrices, epoxy, between them, which showed satisfactory results to increase hardness, if it has a good matrix interface/QC in the surface properties [21]. Polyethylene composites of high molecular mass with QC particles have been tested as an application for acetabulary prostheses, in which the authors ANDERSON et al (2002), show a good tribological evaluation for the composite compared to the pure polymer, among other studies [22].

Several techniques can be used in the processing of films and/or composite coatings, among them we can mention dip coating, Langmuir-Blodgett, spin coating [23–25]. Rotation coating, or spin coating is a technique that stands out for being used to manufacture polymeric films, composites, home cells and electronic devices, in other words, a variety of products. This technique allows the deposition of the material in solution on a substrate that is rotating and, as the solvent evaporates, the film and/or coating is formed, giving it a uniform thickness [25–28].

As can be seen, polyamides and quasicrystalline loads (QC) present, in isolation, interesting characteristics and applications for the industrial environment, however, little is known about the interaction between them, technological development and the various potential applications that can be generated by combining both, in the development of a new composite. Nor are there any studies on the feasibility of the spin coating technique for the production and processing of coatings using these materials.

Thus, the differential of this work consists in the development, characterizations, and analysis of the thermal and surface properties of polyamide 6/Quasicristal composite coatings (PA6/QC) making use of a processing technique slightly used for this type of composite. Since it is part of the science and engineering process to use existing resources in a combined way, thus enabling studies, development, and innovation for future advances [5]. For this purpose, a polymeric solution of PA6/QC was developed, which was used for the development of composite coating via Spin coating techniques. The composite coatings were characterized by thermogravimetric analysis (TG), differential scanning calorimetry (CSD), average roughness (RA), contact angle, surface energy and scanning electron microscopy (SEM). Therefore, the study presented here makes room for future research regarding possible applications of this composites.

2. Materials and methods

2.1. Materials

Polyamide 6 was used, with a density of 1.13 g cm$^{-3}$ and absorption of 2%–4%, already transferred under these conditions by Professor Amanda Leite of the Federal University of Rio Grande do Norte (UFRN). We also used the quasicrystalline alloy (QC), with recycled scrap Al, nominal composition Al$_{62.2}$ Cu$_{25.5}$ Fe$_{12.3}$, a distribution of particles ranging between 0.1 and 30 μm and with average particle diameter of 6.33 μm, manufactured at the Rapid Solidification Laboratory (LSR) at the Federal University of Paraíba (UFPB).
2.2. Quasicrystalline alloy manufacturing and obtaining powder

The precursor components of the alloy in the atomic composition of Al$_{62.2}$ Cu$_{25.5}$ Fe$_{12.3}$ were weighed and then fused, in an induction oven, under an atmosphere of argon gas. Subsequently, they were submitted to thermal treatments, in a controlled atmosphere of argon, in the Na8000 Radiant oven, by the Analogica brand, for homogenization of the quasicrystalline phase in the alloy. Then, it was ground, in a planetary sphere mill, thus obtaining the quasicrystalline powder, a process similar to the one used by Dos Santos Barros, T P et al 2019 [21].

2.3. Manufacture of Polyamide composite coating 6/Quasicrystal

The manufacture of the coating consisted of three steps: production of polyamide 6 solution (PA6), followed by the production of polyamide 6/quasicrystal solution (PA6/QC) and manufacture of pa6/QC composite coating.

To produce PA6 solution was weighed 2 g of PA6 and 15 ml of formic acid, this material was solubilized for 2 h, under magnetic agitation to form the base solution, a procedure similar to the process used by Song J et al [2].

Then, to produce PA6/QC solution, the powders of quasicrystalline particles, previously weighed, in the percentage proportions of 0% of, 1%, 3% and 5% of (%Vol. QC), table 1, were added to 5 ml of the base solution of PA6 and magnetically agitated for another 15 min, each percentage proportion, thus forming the PA6/QC solution for coating manufacturing.

The PA6/QC solution was deposited, using a pipette, in the substrate, which was rotating, at 1200 rpm, through the spin coating technique. After about 1–1.5 min, the solvent was removed, based on the works of Kudyukov, E V et al 2020 and Sahu, N et al 2009 [29, 30]. Therefore, the composite coating of PA6/QC was manufactured, with an average thickness of 0.1 mm, figure 1.

2.4. Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) is a technique that allows the observation of morphologies and sample structure [31]. In which the samples were metallized with gold, SHIMADZU IC50 METALLIZER, using 4 mA chain, then the SSX 550 SUPERSCAN SHIMADZU equipment was used, with a focal length of 12 mm and voltage between 8–20 kV to visualize the morphology of the composites.

2.5. Thermogravimetric analysis (TGA)

Through TG it is possible to monitor losses and/or mass gain of the samples as a time or temperature function [32]. For this work, the composite coatings were submitted to thermogravimetric analyses (TG), which were performed in a SHIMADZU MODEL DTG 60H device, using 3–5 mg of sample in alumina crucible under nitrogen atmosphere (50 ml min$^{-1}$), and heating from 25 °C to 700 °C with heating rate of 10 °C min$^{-1}$.

Table 1. Percentage by volume of quasicrystal in solution.

| Percentage by volume of quasicrystal (%) |
|-----------------------------------------|
| 0                                       |
| 1                                       |
| 3                                       |
| 5                                       |

![Figure 1. Macroscopic images of polyamide 6 composite films/QC via Spin-Coating technique. (a) 0% QC (b) 1% QC, (c) 3% QC and (d) 5% QC.](image-url)
2.6. Differential scanning calorimetry (DSC)

Composite coatings also underwent differential scanning calorimetry (DSC) analysis, in which a DSC 60 PLUS SHIMADZU device was used, using 3–5 mg of alumina crucible sample under nitrogen atmosphere (50 ml min⁻¹) and temperature range from 25 °C to 250 °C, with heating rate of 10 °C min⁻¹. Furthermore, through DSC it was possible to determine at melting temperatures (temperatures of endothermic peaks 1 and 2, Tm1 and Tm2, respectively), fusion enthalpy (areas of endothermic peaks 1 and 2, ΔHm1 and ΔHm2, respectively), crystallization temperature (exothermic peak temperature, Tc), crystallization enthalpy (exothermic peak area, ΔHc), and degree of crystallininess (Xc) (which was calculated using equation (1)) [33, 34].

\[
X_c = \left( \frac{\Delta H_{m1} + \Delta H_{m2}}{(1 - \beta) \cdot \Delta H_{m2}^p} \right) \times 100
\]

Where, \( \beta \) represents the weight fraction of QC and \( \Delta H_{m2}^p \) represents the average melting enthalpy \( \Delta H_{m(\alpha)}^p \) and \( \Delta H_{m(\gamma)}^p \), and results in 240 J g⁻¹ [35, 36].

2.7. Roughness

Roughness represents small cracks and protrusions, which are found on surfaces, being one of the surface characteristics of the materials that directly influence the wettability of a solid [21]. So that by modifying roughness it is also possible to change the wettability, and the higher the roughness, the higher the wettability, according to the studies of homogeneous wettability [37].

For the analysis of average roughness of the samples, the characterization of Profilometry was performed, in which we used the optical equipment contactless, CCI MP, TAYLOR HOBSON, ENGLAND, brand AMETEK. The samples were positioned on a glass plate and the optical reader went through 5 different and equidistant points of each sample analyzed, this optical reader was directly connected to a computer and as the reader traveled over the surface at these 5 points, roughness data (Ra) in \( \mu m \) were generated, which were sent to the computer to create an average of the values in each roughness point, therefore it was possible to identify the average roughness of the samples, table 4.

2.8. Contact angle and wettability

Wettability represents the easiness of a solid to be wetted by a liquid. So the ease or difficulty of wetting will depend on the solid/liquid interfacial forces. Considering the physical wettability, which is determined through the contact angle formed by the liquid in the solid. The measurement of these contact angles indicates the wettability in the solid/liquid interaction. Where angles smaller than 90° indicate high wettability, on the other hand, contact angles greater than 90° represent low wettability [21, 22].

For this work, the contact angle and wettability analysis were used to evaluate the surface characteristics of hydrophobicity, oleophilicity. For this assay, three fluids were used, distilled and saline water, and oil, with drops of 0.50 \( \mu m \). Three measurements were performed in each sample, at a controlled temperature of 23 °C and using the CAMERA Phantom CONTROL (PCC) 2.2 and its Drop Image Capture Software. Next, version 4.5 of Surftens software was used, for the measurements of the contact angles, making use of 5 measurements of the height and dimension of the droplet base for each sample.

2.9. Surface energy

Surface energy can be defined as the relationship between the solid and liquid energies [5]. To perform the analysis of surface energy of the samples and predict the surface energy of the composites (\( \gamma_s \)) equations (2) and (3) were used \( \gamma_s \).

To calculate \( \gamma_s^d \) (dispersive component of solid) and \( \gamma_s^p \) (polar component of solid) equation (2) was used. For the calculations, we used the values of the contact angles from the wettability assay and the literature values for the polar components of the liquid (\( \gamma_L^p \)), liquid dispersive (\( \gamma_L^d \)) and the liquid’s surface energy (\( \gamma_L^s \)) [35, 36]. Then, through equation (3), which represents the sum of the dispersive components of solid (\( \gamma_s^d \)) and polar of solid (\( \gamma_s^p \)), values found through equation (1), it was possible to calculate the surface energy value of composite coatings (\( \gamma_s \)) [21].

\[
\frac{\gamma_L(1 + \cos \theta)}{2\sqrt{\gamma_L^d}} = \sqrt{\gamma_s^d} + \sqrt{\gamma_L^d \gamma_s^p}
\]

\[
\gamma_s^d + \gamma_s^p = \gamma_s
\]
3. Results and discussions

3.1. Scanning electron microscopy (SOW)

The QC particles, figure 2, are well distributed in the matrix, represented by the lighter regions, and intensified as the loads are increased [2]. It is possible to infer the feasibility of the Spin Coating technique for the manufacture of this type of composite coating, figure 1. It is also observed the formation of some quasicrystalline particles (QC) agglomerates figures 2(c) and 2(d), this may be associated with low average particle diameters, value of 6.33 μm, and wide particles distribution, ranging between 0.1 and 30 μm, thus contributing to these agglomerates regions, in addition to the increase of QC volumetric fraction of the samples, figure 2(d). Fact also presented in other studies in which epoxy resin matrix was used [21]. It was also observed the presence of some pores, which may have been caused due to the detachment of QC particles at the time of the solvent evaporation, similar behavior occurs with PA6 composites and clayey materials [38, 39].

Visually, it is observed that the average grain sizes of the composites had a tendency to increase, with the addition of quasicrystalline loads, when compared to the grain size of pure polyamide 6, figures 2 to and 2(d), corroborating the results of the CSD curves and the values of the crystallinity degree, in which there is a change in the crystalline form of Polyamide 6 with the addition of QC, figure 4, and decrease in the crystallinity degree of composites with the addition of QC, table 2 and 3. Thus, it was possible to observe that the addition of QC influences both the morphology of the samples and their crystalline BP6.

3.2. Thermogravimetric analysis (TGA)

Figure 3 presents the thermal analysis curves and their derivative (TG and DTG), respectively, in which it is possible to observe the mass variation of the samples in relation to temperature.

You can see that all samples showed mass loss. Since this mass loss is related to PA6 degradation, and polymeric materials have high thermal sensitivity, due to the type of chemical bond they have, in relation to other materials [21, 31]. The mass loss of the samples 0% QC and 1% QC were more prominent in relation to the other samples, figure 3(b), starting at a temperature around 350 °C and ending around 520 °C. This is due to the low percentage of QC in the samples, so that these temperatures correspond to the beginning and end of the
thermal degradation of polyamide, because the pure QC will present mass loss at temperatures higher than 500 °C \[21, 35, 36\]. For composites with higher volumetric percentages of QC, 3%QC and 5%QC, there was a decrease in mass loss, especially the sample with 5%QC, which presented a residual percentage of mass greater than 50%, figure 3(a), curve in green. This behavior is due to the presence of a higher percentage of quasicrystal

Table 2. Fusion parameters obtained in the first heating and cooling cycle for polyamide 6 as a function of the addition of quasicrystals.

| Percentage by volume of QC (%) | Tm1 (°C) | Tm2 (°C) | ΔHm1 (J g⁻¹) | ΔHm2 (J g⁻¹) | ΔHm1 + ΔHm2 (J g⁻¹) | Xc (%) | Tc (°C) | ΔHc (J g⁻¹) |
|-------------------------------|----------|----------|--------------|--------------|----------------------|--------|--------|------------|
| 0                             | 206.960  | 218.050  | 18.790       | 40.970       | 59.760               | 24.900 | 186.960 | −58.950    |
| 1                             | 209.680  | 219.760  | 25.560       | 35.570       | 61.130               | 27.370 | 185.320 | −47.020    |
| 3                             | 203.290  | 214.970  | 1.270        | 9.10         | 10.370               | 5.290  | 177.510 | −17.520    |
| 5                             | 203.810  | 214.300  | 1.820        | 9.430        | 11.250               | 6.440  | 175.110 | −23.970    |

Table 3. Melting parameters obtained in the second heating cycle for polyamide 6 as a function of the addition of quasicrystals.

| Percentage by volume of QC (%) | Tm1 (°C) | Tm2 (°C) | ΔHm1 (J g⁻¹) | ΔHm2 (J g⁻¹) | ΔHm1 + ΔHm2 (J g⁻¹) | Xc (%) |
|-------------------------------|----------|----------|--------------|--------------|----------------------|--------|
| 0                             | 211.130  | 217.270  | 25.810       | 26.290       | 52.100               |        |
| 1                             | 212.130  | 218.340  | 18.820       | 22.500       | 41.320               | 18.500 |
| 3                             | —        | 214.970  | —            | 13.430       | 13.430               | 6.850  |
| 5                             | —        | 214.300  | —            | 23.940       | 23.940               | 13.700 |
loads in the samples and the fact that the quasicrystal has high density in relation to PA6, in addition to thermal degradation at temperatures greater than 500 °C, thus resulting in this decrease in mass loss in relation to samples of 0%QC and 1%QC, with lower percentage of QC. Not to mention that quasicrystal particles can act as a physical barrier that restricts the mobility of the polymer chains of the PA matrix. Sang, L et al reported that an oxidative layer on the surface of CF/PA6 composites, subjected to a thermooxidative aging process, acted as a barrier to prevent oxygen from penetrating deeper into the internal mass of the composites, and consequently slowed the volatilization of low molar mass molecules, protecting PA6 from further oxidation. This allows us to infer that the increase in quasicrystalline loads contributes to the thermal stability of PA6.

3.3. Differential scanning calorimetry (DSC)

The presence of two endothermal peaks was verified during the heating cycles, figure 4. The first endothermic peak is related to fusion of the crystalline form γ while the second endothermic peak corresponding to fusion of the crystalline form α. It is possible to notice that with the increase in the content of quasicrystalline loads, there is the disappearance of the first peak, γ crystalline form, this is due to interaction of the quasicrystal crystalline particles with polyamide structure, a result similar to other studies with ceramic loads.

In table 2 and 3, it was also possible to observe that there was a reduction in the degree of the samples crystallinity as the percentage of quasicrystal increased for the two heating cycles, corroborating the morphology of the samples, figure 2 and presenting results similar to other composites. Thus, it can infer that the presence of quasicrystal influences the crystallineness of polyamide 6.

3.4. Roughness

In table 4 it is observed values of the samples average roughness (Ra) of pure polyamide 6 and composites, together with their respective standard deviations. The increase in the percentage of quasicrystalline loads causes a decrease in the average roughness of the samples (Ra). This trend may be related to the low average size of quasicrystalline particles, an average diameter of 6.33 μm, together with the effectiveness of the Spin Coating technique, using the polymer in solution, allowing quasicrystal particles to occupy the interstitial spaces of Polyamide 6, thus reducing the average roughness of the samples (Ra). It was also observed the considerable increase in the standard deviation of the sample of 1% of QC compared to the other samples, which may have
been caused by the samples’ low percentage of QC, forming clusters in some regions to the detriment of others [38, 45].

3.5. Contact angle and wettability

Figure 5 shows images of the contact angles formed between solid and distilled water (a), saline water (b) and oil (c) in samples of 0% QC, 1% QC, 3% QC, and 5% QC.

Table 4. Roughness analysis of the sample of 0% QC and composites with 1% QC, 3% QC and 5% QC with quasicrystalline loads.

| Volume of QC (%) | 0   | 1   | 3   | 5   |
|------------------|-----|-----|-----|-----|
| Average Roughness (μm) | 1,011 | 0,984 | 0,822 | 0,789 |
| Standard deviation  | 0,122 | 0,405 | 0,004 | 0,005 |

Figure 5. Images of the contact angles formed between solid and distilled water (a), saline water (b) and oil (c) in samples of 0% QC, 1% QC, 3% QC, and 5% QC.

Table 4. Roughness analysis of the sample of 0% QC and composites with 1% QC, 3% QC and 5% QC with quasicrystalline loads.

been caused by the samples’ low percentage of QC, forming clusters in some regions to the detriment of others [38, 45].

3.5. Contact angle and wettability

Figure 5 shows images of the contact angles formed between solid and distilled water (a), saline water (b) and oil (c). And to improve visualization, the values of the wettability contact angles are represented in figure 6. In which it is possible to observe, both for distilled water and for saline water, there is an increase in the contact angles for the samples of 1% QC, 3% QC and 5% QC in relation to the sample of 0% QC with the increase of the percentage of quasicrystalline load. This behavior is related to the mechanism of liquid/solid interaction and low surface energy inherent to quasicrystalline alloys, together with the decrease in the average roughness of the samples (Ra), which prevent the liquid from spreading through the solid [21, 38, 44, 45]. For the sample 5% QC the contact angles have values lower than the sample of 3% QC. This behavior is associated with the presence of pores in the samples of 5% QC, figure 2(d). Thus, as polyamides 6 are polymeric materials with high affinity for water, it can be understood that the increase in the percentage of quasicrystalline load in the polyamide matrix 6 was responsible for the increase in polymer hydrophobicity in terms of the use of distilled water and saline water [46, 47]. On the other hand, for the wettability with oil, it was possible to observe that there is a reduction in the contact angle of the samples as the percentage of quasicrystalline loads increases. This may be associated with the fact that the oil acts as a self-lubricating for quasicrystalline particles [21]. Not to mention that, in some atmospheric conditions and especially in composites, quasicrystals behave closer to Teflon than to a metal, with low surface energy and low polarity [17, 18, 21]. Thus, causing hydrophobic and ophilolic behavior for composites. Beyond
3.6. Surface energy

Table 5 shows the estimated results for the values of the polar ($\gamma_p^g$) and dispersive ($\gamma_d^g$) components together with the surface energy ($\gamma_g$) of pure Polyamide 6 and composite coatings, calculated using equations (1) and (2).

From the observed values, there is a decrease in the surface energy of the solid, with the percentage addition of quasicrystalline loads. This behavior corroborates the fact that quasicrystalline loads present characteristics of low surface energy and polarity and as their percentage increases within the composite the surface energy of the solid decreases, behavior that also influenced the values of contact angles and wettability of the samples and observed in other studies [17, 18, 48, 49]. The surface energy value was higher than the sample of 3%QC, which for 5%QC, this may be associated with the greater number of pores and agglomerates present in the sample of 5%QC, figure 2(d) [21]. Thus, it can be assumed that quasicrystalline particles contribute to reduce the surface energy value of the samples, influencing their wettability.

4. Conclusions

The composite coatings showed good dispersion of quasicrystalline particles throughout the matrix, despite the presence of some pores and agglomerates, which allows us to consider the feasibility of the Spin Coating technique for manufacturing new types of composites with quasicrystalline loads. The addition of quasicrystalline loads caused greater thermal stability for the composites, acting as a barrier of degradation of the polyamide 6 matrix. In addition, quasicrystalline loads modify the crystalline ness of the polyamide 6 matrix, causing a reduction in the degree of crystalline content of the composites. The reduction of the average roughness (Ra) and the low surface energy of the solid ($\gamma_g$) with a percentage increase of quasicrystalline load, influenced the wettablity and values of the contact angles, causing hydrophobic and oleophilic behavior for the composite coatings. It is possible to conclude that the Spin coating technique is feasible to produce composite coatings with quasicrystalline loads and polymeric polyamide 6 matrix, causing relevant modifications in its surface and composites thermal properties, which leads us to consider further studies regarding interaction and new applications for these coatings.

**Table 5.** Values of polar ($\gamma_p^g$), dispersive ($\gamma_d^g$) and surface energy ($\gamma_g$) components of pure polymer (0%QC) and composites with 1%, 3% and 5%QC ($\gamma_g^{1,3,5}$).

| Volume of QC (%) | $\gamma_p^g$ | $\gamma_d^g$ | $\gamma_g$ |
|------------------|--------------|--------------|------------|
| 0                | 45,630       | 22,300       | 67,930     |
| 1                | 29,720       | 24,510       | 54,230     |
| 3                | 18,760       | 25,500       | 44,260     |
| 5                | 25,380       | 24,730       | 50,120     |
Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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