Microwave dewaxing applied to the investment casting process

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ARTICLE INFO

Article history:
Received 2 February 2008
Received in revised form 5 July 2008
Accepted 12 July 2008

Keywords:
Dewaxing
Investment casting
Microwave
Wax pattern

ABSTRACT

Autoclave processing is commonly used nowadays for dewaxing in the investment casting process. However, since the use of microwave is steadily growing in industrial processes and the wax interacts with the electromagnetic energy of the microwaves, the present work studies the possibility of carrying out dewaxing via microwave. The wax (mineral wax, vegetable resin, low molecular-weight polymer and anti-oxidant) used in this work was prepared by melting in an oven equipped with a mineral oil bath. The chemical and structural stability of the wax were monitored throughout 12 simulated dewaxing cycles, via various analyses, namely, volumetric expansion, hardness, Fourier transform infrared spectroscopy, ultraviolet–visible spectrophotometry, differential scanning calorimetry and viscosity. The results showed that microwave dewaxing is viable, significantly decreasing the incorporation of dirt and water, which is inevitable in the autoclave dewaxing process.

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1. Introduction

The modeling wax pattern used in the investment casting process is a relatively complex mixture of vegetable, mineral and man-made waxes, natural and synthetic resins, organic fillers, additives, and, sometimes, water (Piwonka et al., 2000). The performance of the wax is monitored by the industry so that it may withstand the demands of the various stages of the investment casting (Wolff, 2002), namely, manufacturing cycle of the model, ability to reproduce in details the injection die, physical properties at green suitable to resist handling during assembly of the clusters, good adherence to the ceramic and high-dimensional stability throughout the drying process (Tascioglu and Akar, 2003).

The performance of the wax is usually assessed by evaluating properties like melting point, volumetric expansion, ash content, filler content, resistance to oxidation, chemical inertia, tendency to phase separation, mechanical resistance, tendency to cavitation, plasticity, elasticity, hardness, viscosity, creep resistance, impact strength, welding and welding line characteristics (Sabau and Viswanathan, 2003).

In investment casting, dewaxing has been commonly carried out in industrial autoclaves, in which the molds containing the wax models are placed, being subsequently melted under wet/warm conditions (high temperature and pressure) (Olefin, 2004). The wax originated from this process presents dirt and water, demanding an extra processes, i.e., purification, for its reuse (Bleier and Kukla, 2002).

The wax used in the investment casting process includes materials with dielectric properties, which are able to be heated by microwave absorption. In fact, the use of microwave heating in industrial processes has been growing steadily due to associated advantages regarding minimization of environmental impact, selective and volumetric heating of the
materials, and better product quality (Al-Harahsheh and Kingman, 2004). Indeed, microwave is found in food processing, polymer (Clark and Sutton, 1996; Ku et al., 2001) and ceramic (Bykov et al., 2001) processing, materials synthesis and laboratory procedures (Ku et al., 2002; Benitez et al., 2007; Banik et al., 2003). There are a few published reports in the literature on the influencing factors, advantages and uses of microwave energy for the investment casting process, as in (EPRI, 1993; Anon., 1976; Liu et al., 2007) but this subject still lacks more investigative work.

In this context, this work evaluates the viability of replacing an autoclave by a microwave oven for dewaxing. Even though the latter is a more costly technology, it has the potential of allowing time and energy savings and higher product quality. The study is based on the comparative evaluation of the chemical and structural characteristics and recyclability of a modeling wax after repetitive dewaxing cycles via autoclave and microwave processing.

2. Materials and methods

The wax used in this work was prepared by melting in an oven equipped with mineral oil bath, mechanical stirring and temperature control, following: (i) melting of the macro (18%, w/w) and micro (24%, w/w) paraffin, from Petrobras, along with the polyethylene (LDPE) wax (15%, w/w), from Megh Ceras e Aditivos, at 120–130° C; (ii) addition of ethylene–vinyl acetate copolymer (EVA) (2.5%, w/w), from Polietilenos Uniwax at 120–130 mesh sieves, followed by water removal, i.e., heating of the wax to a pressure at 170–175 °C under stirring until no bubbles could be noticed on the wax surface. This dewaxing procedure via autoclave was repeated for up to 12-dewaxing cycles and, at the end of each cycle, a sample of approximately 30 g was taken.

Another portion of the wax (500 g) was placed on a domestic type of microwave oven (Sanyo) operating under a frequency of 2.45 GHz, a power of 1100 W and for a 20-min period at room pressure (1 atm), reaching a maximum temperature of 120 °C. In this case, no purification treatment was carried out on the resulting wax. Dewaxing via microwave was also repeated 12 times and, at the end of each cycle, a 30 g sample was taken. These samples were tested for:

- Refractive index, in an ABBÉ refractometer (at 80 °C).
- Volumetric expansion (dilation), following the IBER-P-092 standard.
- Shore D hardness (ASTM D 2240-97), in an Instrument MFGCo indenter.
- Viscosity, in a Brookfield viscometer (model DV-II+, Spindle 21).

Besides, Fourier transform infrared spectroscopy (FT-IR), ultraviolet–visible spectrophotometry (UV–vis) and differential scanning calorimetry (DSC) (ASTM D 3418-03) characterizations were carried out using the following equipments: Spectrum 1000 PerkinElmer, Shimadzu spectrophotometer (model 1601PC) and TA Instrument (model 2010), respectively.

The methods used here for the characterization of the waxes may be rigorously applied only to the quantification of pure substances. This is not the case of the multi-component wax used in this work. To overcome this drawback, a standard (virgin) sample was also analyzed, so that reference values and typical UV–vis, FT-IR and DSC profiles were obtained and compared to those from the different waxes after each dewaxing cycle.

3. Results and discussion

Fig. 1 shows the variation of the refractive index with the number of simulated cycles for both dewaxing techniques, namely, autoclave and microwave, along with the respective linear fitting curves. In this figure and the next two ones, the slope of the linear fitting curves is used as a quantitative method to evaluate stability, in a way that the higher the slope, the higher the tendency of the dewaxing method to cause physical–chemical alterations to the wax during continuous cycling of the wax.

Fig. 1 shows that microwave dewaxing is a milder treatment to the wax than the autoclave. A similar finding may be inferred from Fig. 2, i.e., the hardness of the wax is less altered during microwave processing.

Fig. 3 shows the variation of the percentage increase of the volumetric coefficient of thermal expansion of the wax with the number of simulated cycles for both dewaxing techniques. For this comparison, the 60 °C temperature was chosen, since in this temperature the wax is already thoroughly molten. In this analysis, both techniques showed similar results, within the experimental error and therefore the volumetric expansion of the wax was found to be comparably affected by them.

![Fig. 1 – Variation of the refractive index of the wax as a function of the number of cycles for both dewaxing techniques.](image-url)
Fig. 2 – Variation of the Shore D hardness of the wax as a function of the number of cycles for both dewaxing techniques.

From these results, it may be inferred that although non-specific, these tests are sufficiently sensitive to monitor structural (physical) changes in the wax following successive dewaxing cycles. Furthermore, the wax, when removed following a microwave technique, shows less significant structural changes than that found when an autoclave is used. This may have happened because during autoclave dewaxing, the wax undergoes melting in the presence of water steam under high temperature and pressure (170–190 °C and 7.4–7.6 kg cm\(^{-2}\), respectively) inside the autoclave, being then stirred and heated at 120–130 °C for water removal. This procedure may cause the selective removal of components, e.g., the more volatile or hot water soluble ones, or may even cause physical–chemical changes in some of the components of the mixture.

In the following analyses, only the results for the virgin (non-recycled), 6- and 12-cycled waxes will be shown for a better visualization of the variations. Fig. 4 shows the percentage change in volumetric expansion of the virgin, 6- and 12-cycled microwave dewaxed material (Fig. 4a) and the same for the autoclave dewaxed material (Fig. 4b). The microwaved wax, differently from the autoclaved one, appears to show a decrease in dilation with recycling. This may be attributed to non-thermal phenomena caused by the microwaves (Clark and Sutton, 1996), i.e., the successive orientation of the molecules could result in a more packed molecular structure. Thus, expansion could be slightly higher for the autoclaved wax, for which this induced molecular orientation is absent and also because, in this case, thermal vibration could favor a somewhat more chaotic (i.e., more expansive) molecular configuration.

Fig. 5 shows the DSC of the virgin, 6- and 12-cycled microwave dewaxed material (Fig. 5a) and the same for the autoclave dewaxed material (Fig. 5b). Only one major phenomenon can be noticed, which is an endothermic transformation related to melting, i.e., the material appears to behave as a true blend. Besides, the resulting material after microwave dewaxing is more stable than that of the autoclave dewaxing, which again may be a consequence of the more severe process in the latter.

Furthermore, not much difference is noticed between the different cycles for the microwaved wax compared with the autoclave one. In fact, in the microwave-cycled wax, all peaks are close (around 53 °C) whereas the peaks found for the autoclave-cycled wax moved towards slightly higher temperatures (around 57 °C), which may again be an indication that, in the latter, the wax is slightly altered during reprocessing.

Fig. 6 shows the viscosity curves of the waxes after both dewaxing procedures. It can be seen that the wax shows a thixotropic behavior (Qian et al., 1996) and that the microwaves increase wax viscosity. This corroborates the volumetric expansion results, i.e., there are structural changes which increase packing and lead to a greater flowing resistance (Al-Zahrani and Al-Fariss, 1998). The opposite is found for the autoclave dewaxing process, which decreases wax viscosity due to harsh processing conditions (stirring and high temperatures), possibly indicating some degree of thermal cracking.

Fig. 7 shows the ultraviolet spectra of virgin and dewaxed samples from both methods along with the pitch spectrum.
Fig. 5 – DSC results for the virgin, 6- and 12-cycled microwave dewaxed materials (a) and the autoclaved ones (b).

Fig. 6 – Viscosity results for the virgin, 6- and 12-cycled microwave dewaxed materials (a) and the autoclaved ones (b).

Fig. 7 – Virgin, 6- and 12-cycled microwaved waxes and pitch spectra in the ultraviolet range (a), and the same for the autoclaved ones (b).

Analysis of Fig. 7, and specially Fig. 8, shows that, in general, the curves for both dewaxing methods are similar, showing only small variations. The ultraviolet technique is able to detect conjugated bonds of the abietic acids of the pitch and their possible oxidations and cycle-additions (Otter, 1980). However, these reactions were not found to occur in the time length, temperature and pressure conditions to which the waxes have been exposed during either autoclave or microwave dewaxing. In fact, the UV-vis spectrum was only able to detect the pitch, with the maximum absorption occurring around 241 nm. This peak was maintained for the 6- and 12-cycled waxes, thus indicating that the double bonds are still present, i.e., no significant oxidative process has occurred during wax reprocessing.

Regarding the infrared results, the characteristics peaks found in the wax were 2922, 1459 and 724 cm\(^{-1}\), related to the paraffin fraction, and 2922, 1736 and 1282 cm\(^{-1}\), related to the acid wax fraction (pitch) (Socrates, 1994; Pouchet, 1981), which remains present even after wax reprocessing, ratifying the UV-vis results. Besides, no contaminants could be found from the FT-IR spectra.

The absence of wax transformations was expected since the microwave energy that interacts with the wax molecules is not sufficiently high to activate significant degradation reactions under the conditions used in this work (Clark and Sutton, 1996). In addition, the thermo-mechanical process...
4. Conclusions

The hardness, refractive index, and volumetric expansion (dilation) analyses of the wax as a function of the number of reprocessing cycles have shown that:

(i) Although these tests were considered not specific for this aim, they were sensitive enough to monitor wax degradation occurring due to repetitive cycling of the material during autoclave processing.

(ii) The wax which was dewaxed on the microwave shows, in general, less significant physical–chemical changes. Wax melting due to steam under relatively high temperature (170–190 °C) and pressure (7–9 kg cm⁻²) conditions and the subsequent water removal by heating (120–130 °C) and mechanical stirring for the purification of the autoclaved wax may be responsible for the noticed wax alterations, evidencing the advantages of the microwave dewaxing process, which does not require these post-treatment steps.

The wax showed a thixotropic behavior and the viscosity results ratified that microwave dewaxing caused less significant structural changes in the wax. The DSC, FT-IR and UV–vis curves showed only minor differences of varied magnitude and nature and therefore of difficult identification, especially because a multi-component wax was used. Nevertheless, there is again an indication that the microwave dewaxing process is milder and therefore the recycled wax is more chemically and structurally similar to the original one.

In all, although economical aspects and the presence of ceramics were not evaluated here, the findings of this work support the technical viability of substituting the autoclave for the microwave oven for dewaxing, highlighting the advantages of the latter regarding purity of the resulting wax and its structural and chemical integrity, even after repetitive cycling.

REFERENCES