

Recovery of MMA by Depolymerization of PMMA-Based Dental Resins Scraps Rosettes

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ABSTRACT

This work aims to investigate the influence of temperature on the recovery and purity of Methyl Methacrylate (MMA) by depolymerization of Polymethyl Methacrylate (PMMA) dental resins scraps. The experiments were carried out at 350°C, 400°C, and 450°C, 1.0 atm. in laboratory scale, using a borosilicate glass reactor of 125 mL. The density, kinematic viscosity, acidity and refractive index of liquid products were determined experimentally. The chemical compositions of liquid products were determined by GC-MS. The experiments show liquid phase yields between 48.76% and 94.74% (wt.), showing a 1st order exponential growth behavior, increasing with increasing temperature, while that of solid phase (coke) varied between 0.68% and 38.83% (wt.), showing a 1st order exponential decay behavior, decreasing as the temperature increases. The measure liquid products density, kinematic viscosity, and refractive index are close to those of pure MMA at 20°C, showing percent errors between 0.0% and 0.42%, 6.54% and 7.81%, and 0.28% and 0.42% for density, kinematic viscosity, and refractive index, respectively. The GC-MS analysis identified Methyl Methacrylate (MMA) and Ethylene Glycol Dimethyl Methacrylate (EDGMA). The concentrations of Methyl Methacrylate (MMA) varied 94.20% and 95.66% (area), showing a smooth increase with increasing de-polymerization temperature. The pyrolysis of PMMA dental resins scraps has proven to be an effective method to recover high amounts of MMA with high purity.

Key Words: PMMA, Plastics wastes, Pyrolysis, Depolymerization, Recovery of methyl methacrylate.

Introduction

Polymethyl Methacrylate (PMMA) is a polymer compatible with human tissue [1]. PMMA is widely applied in dentistry because of its good biocompatibility, nontoxicity, color and mechanical stability, non-permeability to body fluids, among other properties [1-4].

PMMA is a recyclable polymer [5-9], and hence can be converted to its precursor monomers, namely Methyl Methacrylate (MMA) [6-8]. Pyrolysis is one of the most promising processes to convert polymers, being an alternative for the conversion of PMMA into MMA [6-8]. The reaction products of PMMA pyrolysis produces solid, a gas, and a liquid phase, rich in MMA [6-8]. The liquid phase is composed mainly of MMA [6-8,10-16].

PMMA pyrolysis has been carried out in fluidized bed reactors [6-8,10-12], fixed bed reactors [13,16], conical spouted bed reactors [14], and stirred tank reactors [15,16], in micro [15,17-30], bench [11,13,16], laboratory [8,10,12,14-16,31], and only a few studies were carried out in pilot scale [7,10].

The main parameters that affect the yield and concentration of MMA in the liquid phase are the composition of feed material (pure, filled, and scrap PMMA), reaction temperature, reaction time, and the process scale [7,8,10,16,32]. The main advantages of PMMA pyrolysis is the possibility of using low quality PMMA-based materials [6-8,10,15,16,32-35], and the fact that liquid reaction products are mainly composed

by MMA [6-8,10,12-16,31-32,34-35]. In addition, the liquid reaction products can be easily purified by fractional distillation, in order to obtain pure MMA [6,7,16,35].

In this context, most studies investigated the effect of temperature on PMMA pyrolysis in order to maximize the yield and the concentration of MMA in the liquid phase products [7,8,10,16,31-32]. Despite studies on depolymerization of PMMA by pyrolysis in laboratory, bench, and micro scale [8,10-16,31,32], and pilot or technical scale [6-8,10,34-36], until the moment, no study has investigated the effect temperature on the depolymerization of PMMA-based dental resins rosettes in laboratory scale. Braido et al. [16], investigated the pyrolysis of PMMA-based dental resins teeth in laboratory and semi-pilot scales, as summarized as follows.

Braido et al. [16], investigated the pyrolysis of homopolymer PMMA and PMMA-based dental resins teeth at 250°C, 300°C, 350°C, 400°C, and 450°C reaction times of 60 min, 90 min, and 120 min in a laboratory scale glass reactor ($\phi_{id}=30.0$ mm, H=150 mm, $V_{Reactor}=105$ mL). The experiments in laboratory scale were carried out with 20 g homopolymer PMMA and PMMA-based dental resins scraps. For the experiment in laboratory scale with homopolymer PMMA and PMMA-based dental resins scraps, the yields of liquid phases varied approximately between 9.0% and 89.0% (wt.), increasing with temperature until 400°C, whereas it becomes almost constant between 400°C and 450°C, while those of solids varied approximately between 1.0% and 88.0% (wt.), decreas-

ing exponentially with temperature until 400°C, whereas it becomes almost constant between 400°C and 450°C. The concentrations of MMA in the liquid phase varied between 82.2% and 99.9% (wt.).

This work aims to evaluate the effect of temperature by the pyrolysis of cross-linked PMMA-based dental resins scraps in form of rosettes in laboratory scale using a reactor of 125 mL at 350°C, 400°C, and 450°C, 1.0 atm. on the yield and concentration of MMA in the liquid phase products.

Materials and Methods

Methodology

The applied methodology is summarized by the diagram shown in *Figure 1*. The methodology is described as a logical sequence of ideas, methods, and process to obtain Methyl Methacrylate (MMA) by depolymerization of PMMA based dental resins scraps at 350°C, 400°C, and 450°C, 1.0 atm. in laboratory scale. The experiments carry out to study the influence of temperature on the yield and concentration of MMA in the liquid phase products. The physicochemical properties of liquid phase products were determined experimentally.

Materials

Dentsply Indústria e Comércio Ltd. a (Petrópolis, RJ-Brazil) supplied the PMMA based dental resin residues, as shown in *Figure 2*. The formulation of dental resins produced by polymerization of MMA includes 1.0% (wt.) of TiO₂ and 5.0% (wt.) of Ethylene Glycol Dimethacrylate (EGDMA) [16].

Depolymerization of PMMA

Experimental apparatus and procedures: The experimental apparatus, illustrated in *Figure 3*, contains a cylindrical reactor of 125 mL, a glass nail adapter (condenser), a ceramic heating system of 800 W and a digital temperature control (Therma Model: TH90DP202-000), as described in details elsewhere [37].

By the pyrolysis of PMMA-based dental resins scraps, approximately 50.0 g weighed using a semi-analytical balance (Marte Brazil Model: AL500). After sealing the reactor, the experimental apparatus has been set up. Then, the cooling system is turned on and the water temperature was set at 10°C. Afterwards, the desired heating rate (10°C/min), and temperature (350°C, 400°C, and 450°C) were set-up. The mass of liquid phase and coke were collected and weighed, and the mass of gas computed by difference. The liquid phase products like physicochemical are characterized by density, kinematic viscosity, refractive index and acidity.

Physicochemical Characterization and Chemical Composition of Liquid Phase Products

Physicochemical characterization of liquid phase products: The liquid phase products were physicochemical characterized for refractive index (AOCS Cc 7-25), density (ASTM D4052, 25°C), and kinematic viscosity (ASTM D445/D446, 40°C), and the procedures reported elsewhere [36-41].

Gas chromatography-mass spectrometry of liquid phase products: The compounds within the liquid phase products were identified by CG-MS using a gas chromatograph (Agilent Technologies, Santa Clara, CA, USA, Model: CG-7890B), coupled to MS-5977A mass spectrometer, a SLBTM-5 ms (30 m × 0.25 mm × 0.25 mm) fused silica capillary column, and the temperature program was injector tem-

perature: 290°C; split: 1:20, detector temperature: 320°C and quadrupole: 150°C; injection volume: 1.0 µL; oven: 70°C, 1 min; 10°C/min, 280°C; 13 min. The intensity, retention time, and compound identification were recorded for each peak analyzed according to the NIST mass spectra library [36-41].

Mass Balances and Yields by Depolymerization of PMMA-Based Dental Resins Scraps

The law of conservation of mass was applied within the depolymerization reactor, operating as a closed thermodynamic system, batch mode, yielding the following equations.

$$M_{Reactor} = M_{Feed} \quad (1)$$

$$M_{Reactor} = M_{Coke} + M_{LP} + M_{Gas} \quad (2)$$

Where $M_{Reactor}$ is the mass of pyrolysis reactor, $M_{Feed} = M_{PMMA}$ is the mass of PMMA based dental resins fragments/residues filled into the reactor, M_{Coke} is the mass of coke, M_{LP} is the mass of liquid products, and M_{Gas} is the mass of gas.

Results

Depolymerization of PMMA-Based Dental Resins Scraps

Process conditions, mass balances, and yields of reaction products: *Table 1* and *Figure 4* summarizes the process conditions, mass balances, and yields of liquid, coke, and gas phases by depolymerization of PMMA based dental resins scraps at 350°C, 400°C, and 450°C, 1.0 atm. in laboratory scale, using a reactor of 125 mL.

The experiments show liquid phase yields between 48.76% and 94.74% (wt.), showing a 1st order exponential growth behavior increasing with increasing temperature, while that of solid phase (coke) varied between 0.68% and 38.83% (wt.), showing a 1st order exponential decay behavior, decreasing as the temperature increases. The yields of liquid phase by depolymerization of PMMA [7-8,12,31], and waste PMMA [6-7,12,16,31] to recover MMA is shown in *Table 2*. The yields of liquid phase, depicted in *Table 1* are higher than those reported elsewhere [8,15-16], and very close to those reported in the literature [6-7,12,31]. The yields of liquid phase products obtained at 400°C and 450°C, 1.0 atm. using a fixed bed reactor of 125 mL in laboratory scale is very close to those reported by Kang et al. [12], Popescu et al. [31], and Braido et al. [16], obtained in laboratory scale, proving that experimental data are in agreement to similar studies reported in the literature [12,16,31]. In fact, the high liquid phase reported by Kang et al. [12], is probably due to smaller particle sizes ($\phi_{particle}$) used as feed [12], conferring a higher heat transmission rate. The larger the mean particle size ($\phi_{particle}$) the worse the heat transfer rate, and the lower the conversion of PMMA into MMA. *Figure 5* illustrates from left to right, MMA and liquid phase products obtained by depolymerization of PMMA based dental resins scraps in form of rosettes at 350°C, 400°C and 450°C, 1.0 atm. in laboratory scale respectively. The higher the temperature darker the liquid phase.

Physicochemical properties of liquid phase products: *Table 3* shows the physicochemical properties (density, kinematic viscosity, and refractive index) of liquid phase products. One observes that measured physicochemical properties are close to those of pure MMA, showing that MMA with high purity has been obtained by depolymerization of PMMA based dental resins scraps at 350°C, 400°C, and 450°C, 1.0 atm. in laboratory scale using a reactor of 125 mL.

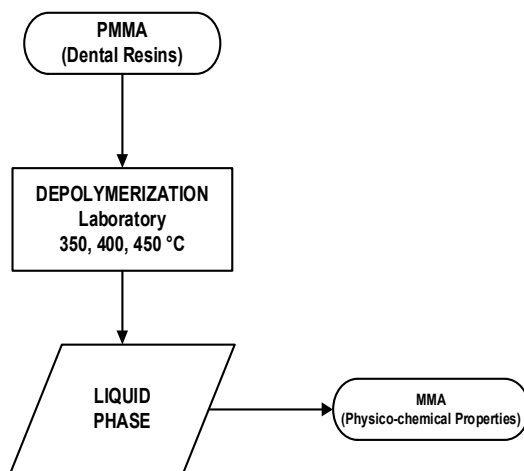


Figure 1: Process diagram by the production of high purity MMA by depolymerization of PMMA-based dental resins scraps in laboratory scale.



Figure 2: PMMA-based dental resins scraps in form of rosettes, discharged by the quality production process control, were used as raw material by depolymerization at 350 °C, 400 °C, and 450 °C, 1.0 atm. in laboratory scale.

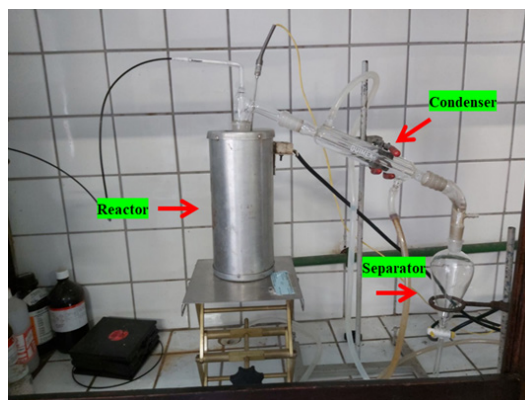


Figure 3: Experimental apparatus (glass reactor in laboratory scale).

Table 1: Process conditions, mass balances, and yields of reaction products by thermal degradation of PMMA based dental resins scraps at 350 °C, 400 °C, and 450 °C, 1.0 atm. in laboratory scale.

Process conditions	Temperature [°C]	Mean ± SD	Mean ± SD
	350	400	450
Mass of feed [g]	30.13	40.03	40.14
Reaction time [min]	60	60	70
Cracking temperature [°C]	350	400	450
Mass of coke [g]	11.7	2.6	0.27
Mass of liquid [g]	14.69	36.45	38.03
Mass of gas [g]	3.74	0.98	1.83
Yield of liquid [g]	48.76	91.06	94.74
Yield of coke [g]	38.83	6.49	0.68
Yield of gas [g]	12.41	2.45	4.58

Table 2: Depolymerization of PMMA [7-8,12,31] and waste PMMA [6-7,13,17], in semi-pilot [6-8,15,16], and laboratory scales [12,31].

Thermal degradation of PMMA and waste PMMA							
	[6]	[7]	[8]	[12]	[15]	[16]	[31]
Yield of liquid phase [%]	96.6	92.13	24.82	96.8	65	66.3	90

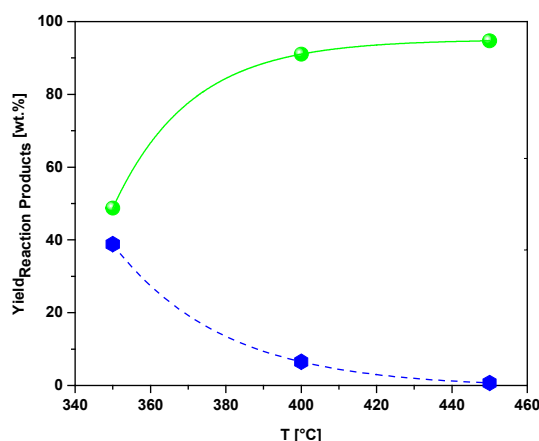


Figure 4: Yield of liquid and gaseous phase products by depolymerization of PMMA-based dental resins scraps in form of rosettes at 350 °C, 400 °C, and 450 °C, 1.0 atm. in laboratory scale. **Note:** (●) Liquid, (●) Solid, (—) $Y_{\text{Liquid}} = -1.23E9 \cdot \exp(-T/20.5) + 35.1, r^2 = 1.0$, (---) $Y_{\text{Solid}} = 6.53E6 \cdot \exp(-T/29.1) - 0.6, r^2 = 1.0$.

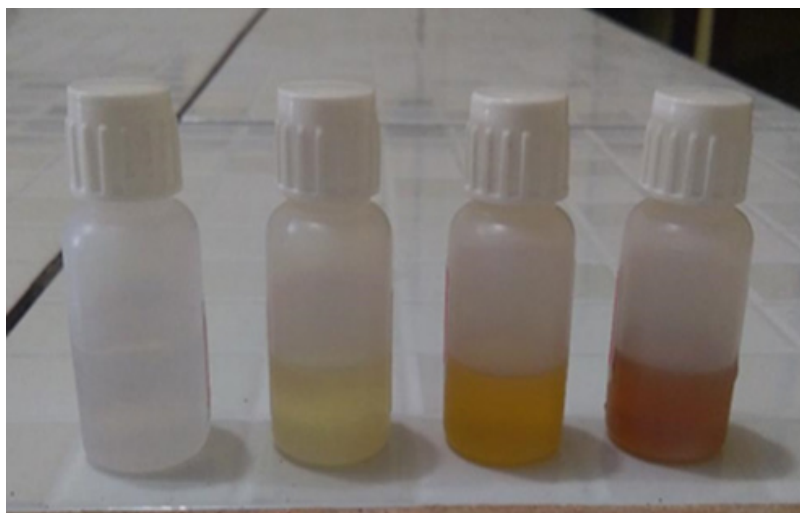


Figure 5: MMA and liquid phase products obtained by depolymerization of PMMA-based dental resins scraps in form of rosettes at 350 °C, 400 °C, and 450 °C, 1.0 atm. in laboratory scale.

Table 3: Physicochemical properties of liquid products obtained at 350 °C, 400 °C, and 450 °C, 1.0 atm. using a reactor of 125 mL, in laboratory scale.

Physicochemical properties	Temperature [°C]			
	350	400	450	References
ρ [g/mL], 30 °C	0.945	0.947	0.949	0.9450 (20 °C)
Refractive index [-], 30 °C	1.42	1.42	1.42	1.414–1.416 (20 °C)
μ [cSt], 40 °C	0.589	0.596	0.594	0.635 cSt (20 °C)

Chemical Analysis of Liquid Reaction Products

GC-MS of liquid reaction products: Figure 6 illustrates the chromatogram of pure Methyl Methacrylate (MMA). The GC-MS analysis identified only 01 (one) peak, Methyl Methacrylate (MMA) with 100.00% (area) and retention time at 3.67 min.

Figure 7 illustrates the chromatogram of liquid phase products obtained by depolymerization of PMMA based dental resins scraps in form of rosettes at 450 °C, 1.0 atm. in laboratory scale. The GC-MS analysis identified only 02 (two) peaks,

Methyl Methacrylate (MMA) with 95.66% (area) and retention time at 3.67 min, Ethylene Glycol Dimethyl Methacrylate (EDGMA) with 4.34% (area) and retention time at 14.00 min. The concentrations of Methyl Methacrylate (MMA) varied 94.20% and 95.66% (area), showing a smooth increase with increasing depolymerization temperature, proving that depolymerization of PMMA based dental resins scraps in form of rosettes at 350 °C, 400 °C, and 450 °C, 1.0 atm. in laboratory scale, produced Methyl Methacrylate (MMA) of high purity. The concentrations of MMA in the liquid phase are according to the literature [6-8,13,16-17,34-36].

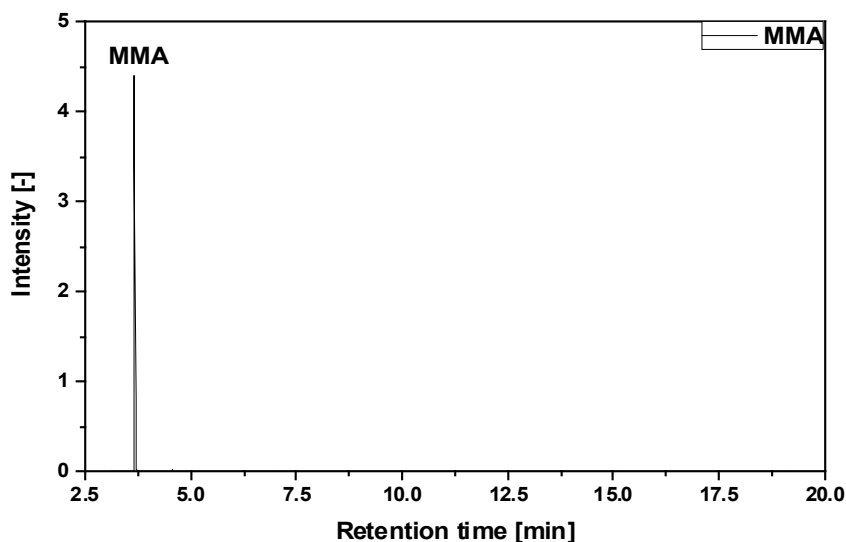


Figure 6: GC-MS of pure MMA.

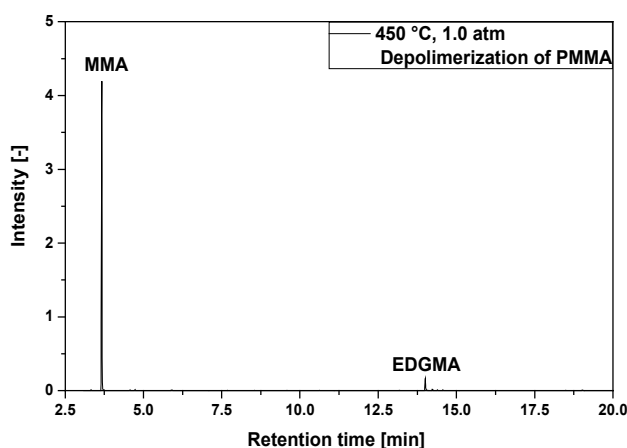


Figure 7: MMA and liquid phase products obtained by depolymerization of PMMA-based dental resins scraps in form of rosettes at 350 °C, 400 °C, and 450 °C, 1.0 atm. in laboratory scale.

Conclusion

The concentrations of Methyl Methacrylate (MMA) varied 94.20% and 95.66% (area), showing a smooth increase with increasing depolymerization temperature, proving that depolymerization of PMMA based dental resins scraps in form of rosettes at 350 °C, 400 °C, and 450 °C, 1.0 atm. in laboratory scale, produced Methyl Methacrylate (MMA) of high purity.

The measure liquid products density, kinematic viscosity, and refractive index are close to those of pure MMA at 20 °C, showing percent errors between 0.0% and 0.42%, 6.54% and 7.81%, and 0.28% and 0.42% for density, kinematic viscosity, and refractive index, respectively. The pyrolysis of PMMA dental resins scraps has proven to be an effective method to recover high amounts of MMA with high purity.

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