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Synthesis, Characterization, Thermal Analysis and Antimicrobial Evaluation of Bis(2-arylamino-2-oxoethyl)terephthalates from the Depolymerization of Post-Consumer PET Bottles

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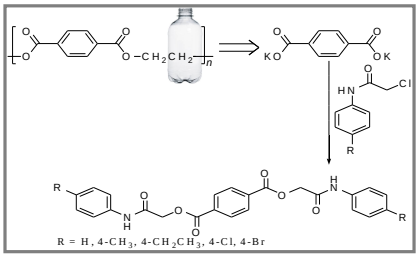
ABSTRACT

*This paper describes the synthesis and characterization of five bis(2-arylamino-2-oxoethyl) terephthalates (**5a-e**), which were obtained in three steps: (i) preparation of potassium terephthalate (**2**) from the depolymerization reaction of post-consumer polyethylene terephthalate (PET) bottles (**1**) at 90% yield; (ii) acetylation reaction of aromatic amines (**3a-e**) with chloroacetyl chloride at 76-95% yield; and (iii) alkylation reaction of recycled potassium terephthalate (**2**) with 2-chloro-N-arylacetamides (**4a-e**) at 30-90% yield. The final compounds (**5a-e**) were characterized by spectroscopic techniques of IR and one- and two-dimensional (for **5a**) NMR, and by mass spectrometry. This work still presents the TGA and DSC curves and the antimicrobial evaluation of the bis(2-arylamino-2-oxoethyl) terephthalates (**5a-e**).*

Graphical Abstract



Highlights

- Five bis(2-arylamino-2-oxoethyl) terephthalates were synthesized through reaction of recycled potassium terephthalate with five 2-chloro-*N*-arylacetamides.
- TGA and DSC curves of terephthalate derivatives were studied.
- Antifungal and antibacterial activity of terephthalate derivatives was evaluated.

Keywords: Recycling, Depolymerization, Terephthalates, Acetamides, Antimicrobial evaluation, Thermal analysis.

INTRODUCTION

Currently, more than 12 million units of PET bottles are produced in Brazil per day. Despite the various applications for post-consumer PET bottles, 45% of the total production is still not reused [**1**]. PET is a condensation polymer and can be submitted to mechanical or chemical recycling [**2**]. The chemical decomposition (chemical recycling) of PET is based on the reversibility of the polymerization reaction, and can be made by the chemical processes of hydrolysis [**3-11**], glycolysis [**12**], methanolysis or aminolysis, and can be catalyzed by acids [**4, 6, 7**], bases [**7-11**] or neutral catalysts [**3, 5, 7, 12**]. Studies show that the depolymerization of PET generally achieves high yields in critical conditions such as the use of concentrated solutions [**4, 6, 11**], high temperatures [**3,5,6,8,12**] and high pressures [**3, 12**]. Some works show the use of aqueous solution of NaOH in mild conditions of concentration (7.5 M), temperature (100 °C) and pressure (1 atm) and lower reaction times [**7, 9, 10**].

Polyethylene terephthalate presents well-defined thermal analytical curves and a well know thermal behavior. TGA curve is used to present the thermal stability. The thermal history effects in the thermal properties of a PET sample from a soft drink bottle are used to demonstrate the effect of different heating/cooling conditions on glass transition, melting, crystallization and crystalline degree using DSC curves [**13**]. Therefore, is interesting to evaluate the TGA and DSC curves of terephthalate derivatives.

Post-consumer PET bottles have been used as raw material to produce nanomaterials [**14**], as well as in the synthesis of new heterocycles with antimicrobial activity (Figure 1) [**15**]. Terephthalic acid derivatives have still been used as precursors in the formation of complexes with some metals, such as rhodium [**16**], vanadium, copper, manganese and titanium [**17**].

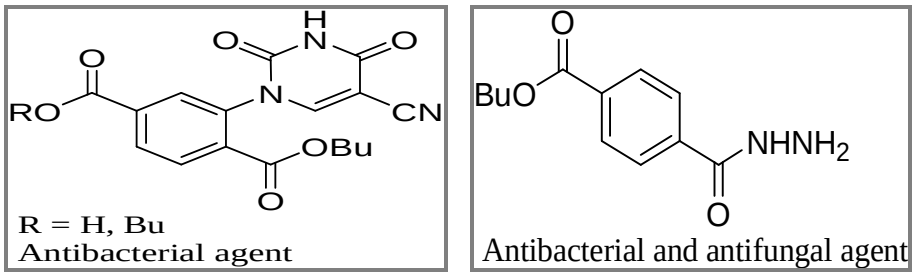


Figure 1. Antibacterial and antifungal agents synthesized from post-consumer PET bottles.

On the other hand, we have seen that 2-chloro-*N*-arylacetamides can be used as antimicrobial agents such as antifungal and antibacterial [**18, 19**]. Our research group has used 2-chloro-*N*-arylacetamides as strategic synthetic intermediates in the reaction with mesoionic 2-(4-chlorophenyl)-4-(4-isopropylphenyl)-3-methyl-1,3-thiazolium-5-thiolate. These mesoionic derivatives were evaluated as new drug candidates due their antifungal activity against several strains of Candida albicans [**20**]. This, combined with the concern to reduce the environmental impact caused by the vast number of post-consumer PET bottles discarded in undue places, led us to study the alkylation reaction of recycled potassium terephthalate with 2-chloro-*N*-arylacetamides, as well as their TGA and DSC curves and evaluation antimicrobial.

MATERIALS AND METHODS

General Methods: All common reagents were purchased from commercial suppliers and used without further purification. The solvents used were HPLC grade, PA or dried according to standard laboratory procedures. The melting points were measure using a MQAPF-302 brand hotplate Microquímica. The melting points were not corrected. FTIR spectra were obtained with a Shimadzu

IRPrestige-21 spectrometer, in the 4000 to 400 cm^{-1} region using KBr pallets and ^{1}H and ^{13}C NMR spectra were obtained with a Bruker and Varian NMR spectrometer, using TMS as internal standard. The mass spectra were registered in an Agilent 6460 Triple Quadrupole ESI-LC/MS/MS. Gas temperature was 300 °C and gas flow was 5 L min^{-1}. Nebulizer remained at 45 psi. The capillary voltage was 3500 V and the shredder was 5 V. Analyte solution was introduced at a flow rate of 0.8 mL min^{-1}. Nitrogen was used as collision and nebulization gas. Molecular ions were detected using the positive and negative mode in the SCAN method. Movel phase used was water:acetonitrile and water:formic acid in 95:5 proportion.

Antimicrobial Activity:

Clinical and Laboratory Standards Institute (CLSI): Document M07-A10.Reference method for dilution antimicrobial susceptibility test of bacteria that grow aerobically; approved standard**.** 10th ed. Clinical and Laboratory Standards Institute: Wayne, Pennsylvania, USA, 2015.

Clinical and Laboratory Standards Institute (CLSI): Document M27-S4. Reference method for broth dilution antifungal susceptibility testing of yeasts; approved standard. 4th ed. Clinical and Laboratory Standards Institute: Wayne, Pennsylvania, USA, 2012.

Clinical and Laboratory Standards Institute (CLSI): Document M38-A2.Reference method for broth dilution antifungal susceptibility testing of filamentous fungi; approved standard. 2nd ed. Clinical and Laboratory Standards Institute: Wayne, Pennsylvania, USA, 2008.

Experimental procedure for synthesis of potassium terephthalate (2): a mixture of crushed post-consumer PET bottles (**1**) (16,31g, 100 mmol) and potassium hydroxide (14,02 g, 250 mmol) was stirred at reflux temperature (140 °C) of anhydrous amyl alcohol (100 mL) for 90 min. Then the mixture was filtered and washed with water (2×100 mL). The aqueous layer was separated and the solvent was evaporated under reduced pressure. The product (**2**) was obtained without further purification. Potassium terephthalate (**2**) in 90% yield; white solid; ^{1}H NMR (200 MHz, D_2O/TMS, 25 °C): δ = 7.80 (s, 4 H, Ph) ppm.

General procedure for synthesis of 2-chloro-*N*-arylacetamides (4a-e): in an ice bath, chloroacetyl chloride (0, 95 mL, 12 mmol) was slowly added dropwise to a stirred mixture of aromatic amine (**3a-e**) (10 mmol) and trimethylamine (1,67 mL, 12 mmol) in anhydrous dichloromethane (10 mL) at 0 °C. The ice bath was removed and the reaction mixture was kept stirring at room temperature for 20 h. Then the mixture was extracted with dichloromethane (10 mL) and washed with ice water (3 × 20 mL). The organic layer was dried with anhydrous magnesium sulfate, filtered, and the solvent was evaporated under reduced pressure. The products (**3a-e**) were purified by crystallization from a solvent mixture of ethanol/water.

2-Chloro-*N*-phenylacetamide (**3a**) in 90% yield; white solid; mp 133-135 °C (from ethanol) (lit.133-135 °C [**20**]); ^{1}H NMR (200 MHz, $CDCl_3$/TMS, 25 °C): δ = 4.16 (s, 2 H, CH_2), 7.15 (t, $J_{H,H}$= 8 Hz, 1 H, Ph), 7.33 (t, $J_{H,H}$ = 8 Hz, 2 H, Ph), 7.52 (d, $J_{H,H}$ = 8 Hz, 2 H, Ph), 8.28 (s, 1 H, NH) ppm.

2-Chloro-*N*-p-tolylacetamide (**3b**) in 95% yield; white solid; mp 182-184 °C (from ethanol) (lit. 182-184 °C **[20]**); ^{1}H NMR (200 MHz, $CDCl_3$/TMS, 25 °C): δ = 2.33 (s, 3H, CH_3), 4.17 (s, 2 H, CH_2), 7.15 (d, $J_{H,H}$= 8 Hz, 2 H, Ph), 7.42 (d, $J_{H,H}$ = 8 Hz, 2 H, Ph), 8.21 (s, 1 H, NH) ppm.

2-Chloro-*N*-(4-ethylphenyl)acetamide (**3c**) in 95% yield; brown solid; mp 140-142 °C (from ethanol) (lit. 140-142 °C [**20**]); ^{1}H NMR (200 MHz, $CDCl_3$/TMS, 25 °C): δ = 1.22 (t, 3H, CH_3), 2.63 (m, 2H, CH_2), 4.17 (s, 2 H, CH_2), 7.18 (d, $J_{H,H}$= 8 Hz, 2 H, Ph), 7.44 (t, $J_{H,H}$ = 8 Hz, 2 H, Ph), 8.23 (s, 1 H, NH) ppm.

2-Chloro-*N*-(4-chlorophenyl)acetamide (**3d**) in 76% yield; gray solid; mp 170-172 °C (from ethanol) (lit. 170-172 °C [**20**]); ^{1}H NMR (200 MHz, DMSO-d_6/TMS, 25 °C): δ = 4.27 (s, 2 H, CH_2), 7.40 (d, $J_{H,H}$= 8 Hz, 2 H, Ph), 7.63 (d, $J_{H,H}$ = 8 Hz, 2 H, Ph), 10.45 (s, 1 H, NH) ppm.

N-(4-Bromophenyl)-2-chloroacetamide (**3e**) in 84% yield; gray solid; mp 184-186 °C (from ethanol) (lit. 184-186 °C [**20**]); ^{1}H NMR (200 MHz, DMSO-d_6/TMS, 25 °C): δ = 4.27 (s, 2 H, CH_2), 7.55 (m, 4 H, Ph), 10.45 (s, 1 H, NH) ppm.

General procedure for synthesis of bis(2-arylamino-2-oxoethyl) terephthalates (5a-e): a mixture of potassium terephthalate (**2**) (1,21 g, 5 mmol), 2-chloro-*N*-arylacetamides (**4a-e**) (10 mmol) and potassium iodide (0,16 g, 1 mmol) was stirred at reflux temperature (100 °C) of DMF (5 mL) for 24 h. The precipitate obtained was filtered and washed with water. The products (**5a-e**) were purified by crystallization from a solvent mixture of DMF/water.

Bis(2-oxo-2-(phenylamino)ethyl) terephthalate (**5a**) in 68% yield; white solid; mp 270 °C (from DMF) (lit. 140-142 °C [**18**]); IR (KBr): ν = 3273 (H-N), 1732 (C=OO), 1668 (C=ON), 1267, 1238, 1116, 1101 (C-O-C and C-N-C), 761 (1,4-disubstituted-Ph), 715, 692 (Ph) cm^{-1}; ^{1}H NMR (400 MHz, DMSO/TMS, 25 °C): δ = 5.00 (s, 4 H, CH_2), 7.08 (t, $J_{H,H}$= 8 Hz, 2 H, Ph), 7.33 (t, $J_{H,H}$ = 8 Hz, 4 H, Ph), 7.61 (d, $J_{H,H}$ = 8 Hz, 4 H, Ph), 8.22 (s, 4 H, 1,4-disubstituted-Ph), 10.27 (s, 2H, NH) ppm; ^{13}C NMR (100 MHz, DMSO/TMS, 25 °C): δ = 63.54 (CH_2), 119.33 (Ph), 123.62 (Ph), 128.85 (Ph), 129.84 (1,4-disubstituted-Ph), 133.41 (1,4-disubstituted-Ph), 138.44 (Ph), 164.77 (C=OO), 165.12 (C=ON) ppm; ESI-MS (N): *m/z* 863.3 [M^++M^+-1], 431.1 [M^+-1].

Bis(2-(p-toluidino)-2-oxoethyl) terephthalate (**5b**) in 89% yield; white solid; mp 262-264 °C (from DMF); IR (KBr): ν = 3261 (H-N), 1734 (C=OO), 1674 (C=ON), 1278, 1255, 1130, 1111 (C-O-C and C-N-C), 812 (1,4-disubstituted-Ph), 723 (1,4-disubstituted-Ph) cm^{-1}; ^{1}H NMR (500 MHz, DMSO/TMS, 70 °C): δ = 2.27 (s, 6H, CH_3), 4.95 (s, 4 H, CH_2), 7.13 (t, $J_{H,H}$= 5 Hz, 4 H, C_6H_4Me), 7.47 (d, $J_{H,H}$ = 5 Hz, 4 H, C_6H_4Me), 8.19 (s, 4 H, 1,4-disubstituted-Ph), 9.96 (s, 2H, NH) ppm; ^{13}C NMR (166 MHz, DMSO/TMS, 70 °C): δ = 20.77 (CH_3), 64.08 (CH_2), 120.27 (Ar), 129.51 (Ar), 130.13 (Ar), 133.20 (Ar), 134.06 (Ar), 136.29 (Ar), 165.22 (C=O), 165.25 (C=O) ppm; ESI-MS (N): *m/z* 919.3 [M^++M^+-1], 459.1 [M^+-1].

Bis(2-(4-ethylphenylamino)-2-oxoethyl) terephthalate (**5c**) in 45% yield; yellow solid; mp 270-272 °C (from DMF); IR (KBr): ν = 3292 (H-N), 1739 (C=OO), 1662 (C=ON), 1276, 1247, 1116, 1105 (C-O-C and C-N-C), 835 (1,4-disubstituted-Ph), 721 (1,4-disubstituted-Ph) cm^{-1}; ^{1}H NMR (400 MHz, DMSO/TMS, 70 °C): δ = 1.15 (t, $J_{H,H}$= 8 Hz, 6H, CH_3), 2.55 (q, $J_{H,H}$= 8 Hz, 4H, CH_2), 4.96 (s, 4 H, CH_2), 7.16 (d, $J_{H,H}$= 12 Hz, 4 H, C_6H_4Et), 7.49 (d, $J_{H,H}$ = 8 Hz, 4 H, C_6H_4Et), 8.20 (s, 4 H, 1,4-disubstituted-Ph), 10.20 (s, 2H, NH) ppm; ^{13}C NMR (100 MHz, DMSO/TMS, 70 °C): δ = 16.15 (CH_3), 28.06 (CH_2), 63.97 (CH_2), 119.86 (Ar), 128.49 (Ar), 130.30 (Ar), 133.87 (Ar), 136.58 (Ar), 139.48 (Ar), 165.22 (C=O), 165.33 (C=O) ppm; ESI-MS (N): *m/z* 1011.4 [M^++M^++^{35}Cl], 523.2 [M^++^{35}Cl].

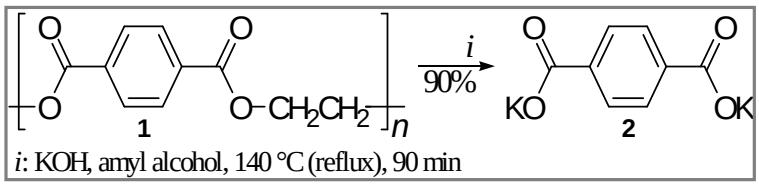
Bis(2-(4-chlorophenylamino)-2-oxoethyl) terephthalate (**5d**) in 90% yield; white solid; mp 302-304 °C (from DMF); IR (KBr): ν = 3257 (H-N), 1732 (C=OO), 1666 (C=ON), 1267, 1246, 1124, 1105 (C-O-C and C-N-C), 829 (1,4-disubstituted-Ph), 723 (1,4-disubstituted-Ph) cm^{-1}; ^{1}H NMR (500 MHz, DMSO/TMS, 70 °C): δ = 4.97 (s, 4 H, CH_2), 7.37 (d, $J_{H,H}$= 5 Hz, 4 H, C_6H_4Cl), 7.62 (d, $J_{H,H}$ = 5 Hz, 4 H, C_6H_4Cl), 8.20 (s, 4 H, 1,4-disubstituted-Ph), 10.19 (s, 2H, NH) ppm; ^{13}C NMR (125 MHz, DMSO/TMS, 70 °C): δ = 64.03 (CH_2), 121.66 (Ar), 127.88 (Ar), 129.09 (Ar), 130.18 (Ar), 133.97 (Ar), 137.77 (Ar), 165.19 (C=O), 165.67 (C=O) ppm; ESI-MS (N): *m/z* 1039.1 [M^++M^++^{35}Cl].

Bis(2-(4-bromophenylamino)-2-oxoethyl) terephthalate (**5e**) in 30% yield; white solid; mp 312-314 °C (from DMF); IR (KBr): ν = 3255 (H-N), 1730 (C=OO), 1666 (C=ON), 1267, 1244, 1124, 1105

(C-O-C and C-N-C), 813 (1,4-disubstituted-Ph), 721 (1,4-disubstituted-Ph) cm^{-1}; ^{1}H NMR (400 MHz, DMSO/TMS, 70 °C): δ = 4.98 (s, 4 H, CH_2), 7.51 (d, $J_{H,H}$= 8 Hz, 4 H, C_6H_4Br), 7.57 (d, $J_{H,H}$ = 8 Hz, 4 H, C_6H_4Br), 8.20 (s, 4 H, 1,4-disubstituted-Ph), 10.42 (s, 2H, NH) ppm; ^{13}C NMR (100 MHz, DMSO/TMS, 70 °C): δ = 63.98 (CH_2), 115.70 (Ar), 121.70 (Ar), 130.32 (Ar), 132.16 (Ar), 133.81 (Ar), 138.26 (Ar), 165.20 (C=O), 165.81 (C=O) ppm; ESI-MS (N): *m/z* 624.9 [M^++^{35}Cl].

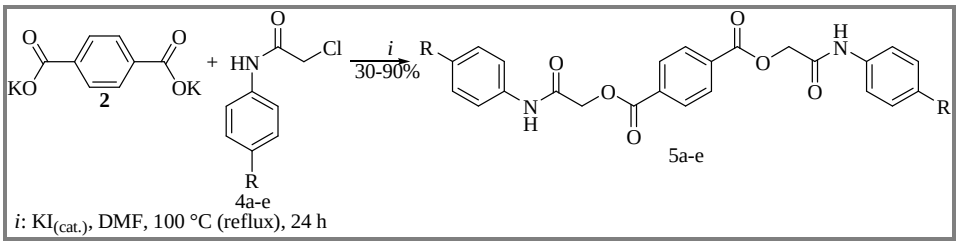
RESULTS AND DISCUSSION

Syntheses: The first step involves the preparation of potassium terephthalate (**2**) (90% yield) through the depolymerization reaction of post-consumer PET bottles with potassium hydroxide under reflux of anhydrous amyl alcohol for 90 min (Scheme 1).



Scheme 1. Preparation of potassium terephthalate (**2**)

The second step consists of the acetylation reaction of aromatic amines (**3a-e**) with chloroacetyl chloride and triethylamine in anhydrous dichloromethane at room temperature for 20 h [**20**]. 2-Chloro-*N*-arylacetamides (**4a-e**) were obtained at 76-95% yield. In the last step, bis(2-arylamino-2-oxoethyl) terephthalates (**5a-e**) (30-90% yield) were synthesized by the alkylation reaction of recycled potassium terephthalate (**2**) with 2-chloro-*N*-arylacetamides (**4a-e**) and potassium iodide as a catalyst in DMF under reflux for 24 h (Scheme 2).

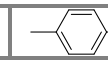


	a	**b**	**c**	**d**	**e**
R	H	CH_3	CH_2CH_3	Cl	Br
Yield (%)	68	89	45	90	30

Scheme 2. Synthesis of bis(2-arylamino-2-oxoethyl) terephthalates (**5a-e**)

IR Spectra: The major change in the IR spectra of the final compounds (**5a-e**) is when the terminal benzene ring changes from unsubstituted (**5a**) to substituted (**5b-e**) (Table 1).

Table 1. Some data from IR spectra

ν (cm^{-1})	H-N	C=OO	C=ON	1,4-disubstituted-Ph	
5a, R = H	3273	1732	1668	761	715 and 692
5b, R = CH_3	3261	1734	1674	812	723
5c, R = CH_2CH_3	3292	1739	1662	835	721
5d, R = Cl	3257	1732	1666	829	723
5e, R = Br	3255	1730	1666	813	721

NMR Spectra: The final compounds (**5a-e**) were characterized by spectroscopic technique of one- and two-dimensional (for **5a**) NMR. The use of two-dimensional spectra was important for complete assignment of signals in the ^{13}C NMR spectrum of the compound **5a**. For example, through ^{1}H-^{13}C

coupling in Heteronuclear Single-Quantum Correlation (HSQC), it was possible to distinguish the aromatic signals (119.33, 123.62, 128.85 ppm for phenyl, and 129.84 ppm for 1,4-disubstituted-phenyl), and through long-distance couplings $^{3\text{-}4}J$ in Heteronuclear Multiple-Bond Correlation (HMBC), it was possible to distinguish the carbonylic signals (164.77 ppm for carbonyl ester, and 165.12 ppm for carbonyl amide) and the quaternary aromatic signals (133,4 ppm for 1,4-disubstituted-phenyl and 138.44 ppm for phenyl).

Mass Spectra: In electrospray ionization mass spectrometry (negative), the compounds **5a-b** exhibited peaks related to the interaction between two molecules $[M^+ + M^+ - 1]$, while the compounds **5c-d** exhibited peaks related to the interaction between two molecules and chloride $[M^+ + M^+ + {}^{35}Cl]$, and the compound **5e** exhibited a peak related to the interaction with chloride $[M^+ + {}^{35}Cl]$.

Thermal Analysis: DSC (melting point and enthalpy data) and TGA (decomposition temperature) curves of terephthalate derivatives (**5a-e**) were studied. Differential Scanning Calorimetry (DSC) is that which accompanies heat changes in the sample, during heating or cooling, relative to an inert material. For example, the DSC analysis for compound **5a** shows that there are two transitions, where the two smaller peaks, both for heating and cooling, are a possible indication of crystal rearrangement (Figure 2).

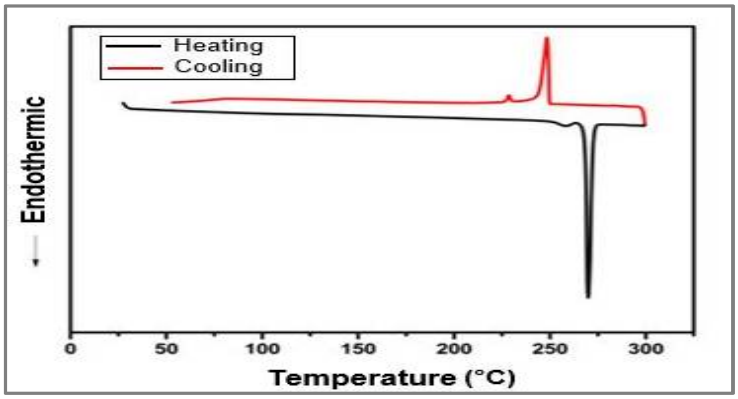


Figure 2. DSC thermogram of bis(2-oxo-2-(phenylamino)ethyl) terephthalate (**5a**).

Thermogravimetric analysis or thermal gravimetric analysis (TGA) is a method of thermal analysis in which the mass of a sample is measured over time as the temperature changes. The TGA curves for compounds **5a** and **5c** show thermal stability, whereas for the other compounds (**5b** and **5d-e**) there are mass losses during heating.

Antimicrobial Evaluation: Compounds **5a-e** were evaluated for their in vitro antimicrobial activity against a panel of microorganisms, including yeasts (*Candida albicans* ATCC 14057, *Candida glabrata* ATCC 90030, *Candida tropicalis* ATCC 750, *Saccharomyces cerevisiae* ATCC 2601 and *Cryptococcus neoformans* var. gattii ATCC 56990), filamentous fungi (*Aspergillus fumigatus* clinically isolated, *Aspergillus niger* clinically isolated, *Aspergillus flavus* clinically isolated, and *Aspergillus terreus* clinically isolated), bacteria (*Bacillus subtilis* ATCC 19659, *Escherichia coli* ATCC 25921, *Klebsiella pneumoniae* clinically isolated, *Pseudomonas aeruginosa* ATCC 9027, *Salmonella typhimurium* ATCC 14028, and *Staphylococcus aureus* ATCC 25923 and alga (*Prototheca zopfii* clinically isolated 401AD) by determining their minimal inhibitory concentration (MIC) methods according to CLSI standards.

Compounds **5a-e** did not show activity against the microorganisms mentioned in the concentration range of 80-0.31 $\mu g.mL^{-1}$. Compound **2** was not evaluated due to it slow solubility in DMSO. The complete methodology utilized for in vitro antimicrobial activity of the compounds **2** and **5a-e** can be found in materials and methods.

APPLICATION

The choice for post-consumer PET bottles as a raw material comes from the possibility of adding value to new substances obtained from recyclable sources, which, in addition, are candidates for antibacterial and antifungal agents, besides allowing the reduction of waste in the environment.

CONCLUSION

In summary, we described a simple route for preparing bis(2-arylamino-2-oxoethyl) terephthalates (**5a-e**) at 30-90% yield through an alkylation reaction of a variety of 2-chloro-*N*-arylacetamides (**4a-e**) with recycled potassium terephthalate (**2**). The thermal analysis showed that only compound **5a** showed congruence between the heating and cooling processes, and just compounds **5a** and **5c** showed thermal stability. The bis(2-arylamino-2-oxoethyl) terephthalates (**5a-e**) did not show significant results against bacteria and fungi in the concentration range of 80-0.31 $\mu g.mL^{-1}$. At the moment, new symmetric and non-symmetric alkylation reactions of recycled potassium terephthalate are underway, as well as new studies of antimicrobial activity.

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REFERENCES

[1]. W. Romão, M. A. S. Spinacé, M.-A. D. Paoli, Poli(tereftalato de etileno), PET: uma revisão sobre os processos de síntese, mecanismos de degradação e sua reciclagem, *Polimeros: Cienc. Tecnol.*, **2009**, 19, 121-132.
[2]. F. Awaja, D. Pavel, Recycling of PET, *Europ. Polym. J.*, **2005**, 41, 1453-1477.
[3]. J.R. Campanelli, M.R. Kamal, D.G. Cooper, A kinetic study of the hydrolytic degradation of polyethylene terephthalate at high temperatures, *J. Appl. Polym. Sci.*, **1993**, 48, 443-451.
[4]. T. Yoshioka, N. Okayama, A. Okuwaki, Kinetics of hydrolysis of PET powder in nitric acid by a modified shrinking-core model, *Ind. Eng. Chem. Res.*, **1998**, 37, 336-340.
[5]. C.-Y. Kao, B.-Z. Wan, W.H. Cheng, Kinetics of hydrolytic depolymerization of melt poly(ethyleneterephthalate), *Ind. Eng. Chem. Res.*, **1998**, 37, 1228-1234.
[6]. T. Yoshioka, N. Okayama, A. Okuwaki, Kinetics of hydrolysis of poly(ethylene terephthalate) powder in sulfuric acid by a modified shrinking-core model, *Ind. Eng. Chem. Res.*, **2001**, 40, 75-79.
[7]. S. D. Mancini, M. Zanin, Influência de meios reacionais na hidrólise de PET pós-consumo, *Polimeros: Cienc. Tecnol.*, **2002**, 12, 34-40.
[8]. A. Ruvolo-Filho, P. S. Curti, Estudo cinético da reação heterogênea de despolimerização do PET pós-consumo em meio alcalino-influência da velocidade de agitação, *Polimeros: Cienc. Tecnol.*, **2006**, 16, 279-285.
[9]. L. D. Souza, M. C .M. Torres, A. C. Ruvolo-Filho, Despolimerização do poli (tereftalato de etileno) - PET: efeitos de tensoativos e excesso de solução alcalina, *Polimeros: Cienc. Tecnol.*, **2008**, 18, 334-340.
[10]. G. Vanini, E. V. R. Castro, E. A. Silva-Filho, W. Romão, Despolimerização química de PET grau garrafa pós-consumo na presença de um catalisador catiônico, o brometo de hexadeciltrimetrilamônio (CTAB), *Polimeros: Cienc. Tecnol.*, **2013**, 23, 425-431.
[11]. T. G. Fonseca, Y. M. B. Almeida, G. M. Vinhas, Reciclagem química do PET pós-consumo: caracterização estrutural do ácido tereftálico e efeito da hidrólise alcalina em baixa temperatura, *Polimeros: Cienc. Tecnol.*, **2014**, 24, 567-571.

[12]. J. R. Campanelli, M. R. Kamal, D. G. Cooper, Kinetics of glycolysis of poly (ethylene terephthalate) melts, *J. Appl. Polym. Sci.*, **1994**, 54, 1731-1740.
[13]. G. Bannach, G. L. Perpétuo, E. T. G. Cavalheiro, C .C. S. Cavalheiro, R. R. Rocha, Efeitos da história térmica nas propriedades do polímero PET: um experimento para ensino de análise térmica, *Quim. Nova*, **2011**, 34, 1825-1829.
[14]. J. O. Alves, C. Zhuo, Y. A. Levendis, J. A. S. Tenório, Síntese de nanomateriais utilizando garrafas PET pós-consumo como matéria-prima, *Tecnol. Metal. Mater.Miner.*, **2012**, 163, 59-63.
[15]. A. M. Fahim, A. M. Farag, G. A. M. Nawwar, PET waste recycling as chemical feedstock: synthesis and antimicrobial activity of new compounds with anticipated industrial use, *J. Applicable Chem.*, **2013**, 2, 502-510.
[16]. D. K. Kumar, A. S. Filatov, M. Napier, J. Sun, E. V. Dikarev, M. A. Petrukhina, Dirhodium paddlewheel with functionalized carboxylate bridges: new building block for self-assembly and immobilization on solid support, *Inorg. Chem.*, **2012**, 51, 4855-4861.
[17]. H. Hosseini-Monfared, N. Asghari-Lalami, A. Pazio, K. Wozniak, C. Janiak, Dinuclear vanadium, copper, manganese and titanium complexes containing O,O,N-dichelating ligands: synthesis, crystal structure and catalytic activity, *Inorg. Chim. Acta*, **2013**, 406, 241-250.
[18]. S. A. Katke, S. V. Amrutkar, R. J. Bhor, M. V. Khairnar, Synthesis of biologically active 2-chloro-*N*-alkyk/aryl acetamide derivatives, *Int. J. Pharm. Sci. Res.*, **2011**, 2,148-156.
[19]. M. Aschale, Synthesis and antimicrobial evaluation of some novel substituted 2-chloroacetanalides, *Int. J. ChemTech Res.*, **2012**, 4, 1437-1441.
[20]. I. N. Peixoto, H. D. S. Souza, B. F. Lira, D. F. Silva, E.O. Lima, J. M. Barbosa-Filho, P .F. Athayde-Filho, Synthesis and antifungal activity against Candida strains of mesoionic system derived from 1,3-thyazolium-5-thiolate, *J. Braz. Chem. Soc.*, **2016**, 27, 1807-1813.