Experimental Study of the Influence of Raw Material Impurities on Yellowness Index of Transesterification Polycarbonate

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ABSTRACT: This article covers a quantitative experimental study on the influence of raw materials and materials of construction impurities on polycarbonate color as expressed by a solution yellowness index. Major impurities in the production process were identified, synthesized, and spiked into the baseline materials. A fractional factorial experimental design was executed to provide some information of the influence of impurities as corresponding to levels similar to those occurring in the manufacturing practice. Analysis of the experimental design showed that isopropenylphenol (IPP), methoxyphenyl benzoate (MOB) and 9,9-dimethylxanthene (DMX) are the main producers of color bodies. A quantitative expression was derived which can be used to predict manufacturing problems with regards to color. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 1348–1356, 2011

Key words: yellowness index; bisphenol A; diphenylcarbonate; polycondensation

INTRODUCTION

Although work in the field of polycarbonates is heading into its sixth decade, a large amount of research is still necessary to fully develop our understanding of this industrially important material. Since the 80s, a great attention has been paid to these transparent polymers due to their intrinsic properties as a substrate for optical data storage media.[1–3] Given the current trends observed in optical data storage systems, it seems that fundamental knowledge on the relationships between structure and product properties will be indispensable for a proper manufacture of the next generation of these materials.

Polymerization processes commonly induce a slight yellow or straw hue in the resins produced. Yellowness index (YI) is a numerical representation of how yellow, a material, is in comparison with a clear water-white standard. Lower YI values indicate greater clarity. The YI of polycarbonate resins is generally about 0.5 to 2.0. This is slightly less yellow than most other commercially available transparent polymers,[4] which typically have YI values of 1.0 to 3.0.

Color is a complex phenomenon. According to Nassau,[5] no less than 15 distinct mechanisms explain the various causes of color ranging from electronic excitations to geometrical and physical optics. Most chemical compounds contain only paired electrons that require very high energies to become unpaired and form excited energy levels; this requires ultraviolet, hence there is no visible absorption and therefore no color. However, in organic molecules, particularly those containing conjugated chains of alternating single and double bonds, e.g., as in the polyenes, the double bonding *pz* orbitals have a variety of excited states. Examples of polyene-type colorants are the orange β-carotene pigment of carrots, the pink carotenoids of flamingos, and the yellow crocin present in saffron.

Polycarbonate manufacturers know that purity of bisphenol A (BPA) and diphenylcarbonate (DPC) is critical to color. Also, the presence of oxygen, residual monomers, remaining catalysts, degraded materials or metals in a resin increases its yellowness. Some of the impurities found in the raw materials are colored such as 4-isopropenylphenol (IPP) but most of the others are not. Metallic impurities come from the habitual materials of construction and traces of catalysts used in the melt transesterification process and also increase YI.

Excessive heat or shear stress during the fabrication processes tends also to increase the YI of a natural resin. To avoid raising the yellowness of the parts, the fabricators should be careful to avoid excessive heat and limit the amount of incorporated regrind material to a recommended maximum level, because regrind (with its own thermal history) tends

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to increase YI. Therefore, the YI value is a proper indicator of the cleanliness of the final polymer.

Normal practice in the manufacture of transparent products is to add a small amount of a blue tint to mask the yellowness. However, the new market specifications impose the need for natural resins without the added blue tint. Therefore, studies to improve manufacturability of these resins are of major interest.[6]

It is well known in the manufacture practice that the impurities in the raw materials, materials of construction, and operating conditions have a definite influence on final YI. However, no quantitative studies have been reported so far. This article is intended to develop a quantitative relationship between the YI in polycarbonate as related to the main impurities content in the raw materials, as well as potential interactions between them which may reveal an intrinsic mechanism of color formation. As a result, this new quantitative relationship could be used to improve manufacturability and establish raw materials specifications.

TABLE I
Impurities Characterization in Reagent Materials

Impurity	Bisphenol acetone (BPA)	Diphenyl carbonate (DPC)
Organic (ppm)		
Phenol	20	550
Isopropenyl phenol (IPP)	40	(–)
o,p-Bisphenol A	2000	(–)
Dimmers	70	(–)
Trisphenol-I	320	(–)
Chroman	80	(–)
spirobiindane (SBI)	30	(–)
Trisphenol-II	1610	(–)
9,9-Dimethylxanthene	90	(–)
Phenyl methyl carbonate (PMC)	(–)	50
2-Methoxyphenyl benzoate (MOB)	(–)	8
Anisol	(–)	40
Metals, (ppm)		
Iron, ppm	100	95
Titanium, ppm	(–)	245
Copper, ppm	50	105
Nickel, ppm	65	55

MATERIALS AND METHODS

Experimental set-up

The experiments consisted in a deliberated contamination of the raw materials with high loads of the most relevant impurities occurring in the raw materials. Then the reaction was conducted following a predetermined temperature profile and finally, YI was determined in solution. Baseline values for major organic and metallic impurities are shown in Table I.

Experiments were carried out in an AIM500 Block Digestion System (Cole Parmer, USA). This device is a high temperature heating block provided with 50 wells which can hold 75 mL tubes. The heating block can go up to 450°C with a set-point accuracy of ±2°C. The temperature in each digestion position is tightly regulated and controlled, ensuring that all the samples in each batch are reacted equally. It can store a temperature program for staged reactions. This device was provided with a self-made header which allowed both nitrogen inertization and vacuum application.

Polymerizations were carried out in 75 mL quartz reactors. Prior experience had shown that there is indeed significant "uncatalysed" reaction in standard borosilicate glass flask due to the presence of trace levels of sodium on the surface.[7] It was also found that switching to quartz reactors the catalytic effect of the reactor surface can be significantly reduced. We also found that catalytic impurities could be introduced from stoppers grease and the borosilicate pipettes used to sample the reaction. The latter effect was suppressed by rinsing the pipettes thoroughly with hydrochloric acid and deionized water. All reactors were washed with concentrated HCl for 1 day prior to polymerization and rinsed with deionized water to take any metal impurities away from their surface.

Materials

A list of the impurities occurring in the raw materials can be found in Figure 1. Some of the organic impurities can be easily obtained from regular suppliers. For instance, BPA, DPC, phenol, and anisole were provided by Sigma Aldrich Química (Tres Cantos, Madrid). Also, phenylmethyl carbonate was supplied by FAR Chemical Inc. (Palm Bay, Florida) and 2-methoxyphenyl benzoate (MOB) was supplied by Leancare Ltd. (Colwyn Bay, United Kingdom). However, most of the remaining organic impurities had to be synthesized according to following procedures.

4-isopropenylphenol

IPP was obtained from 4-hydroxiacetophenone in a two step synthesis involving the addition of a methyl magnesium bromide and the dehydration of the resulting carbinol. IPP is a highly unstable product as it decomposes under storage. Therefore, it had to be synthesized every time "*in situ*" prior to its use.

2′,4‴′-dihydroxy-2,2-diphenylpropane (o,p-BPA)

o,*p*-BPA was synthesized by a Grignard reaction on methyl salicylate as described by Hetper et al.[8]

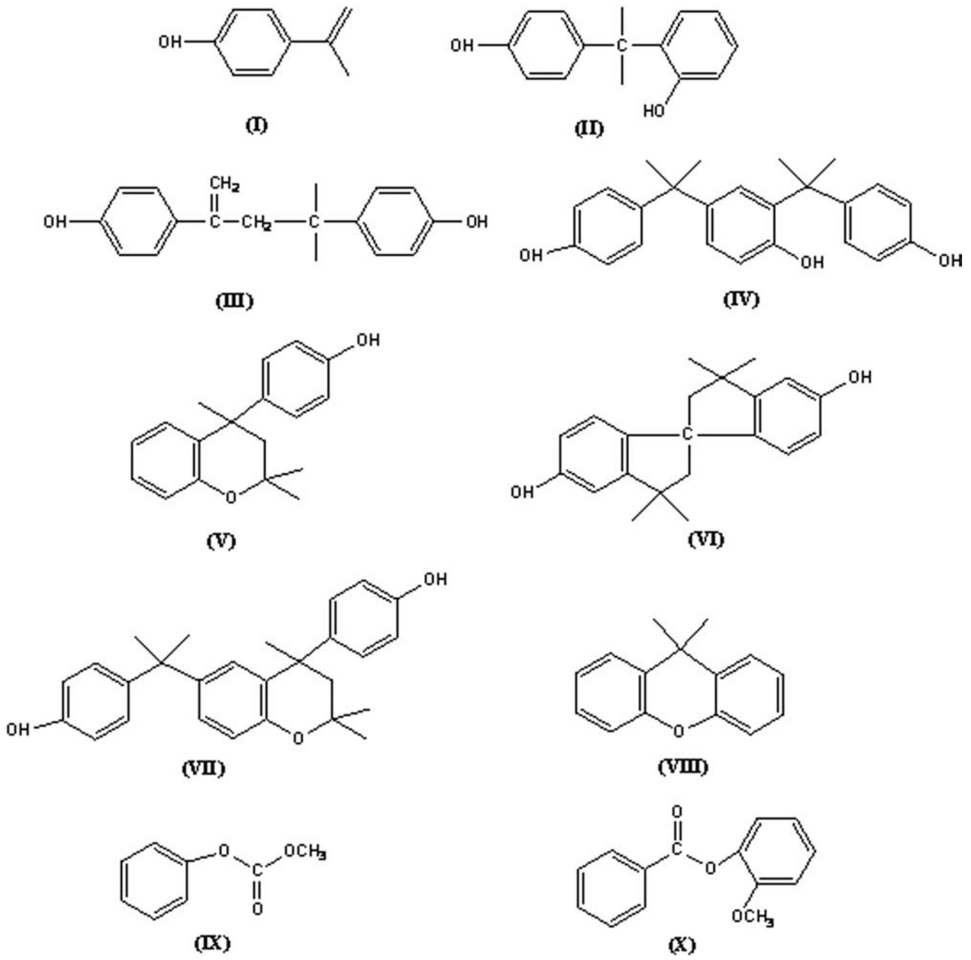


Figure 1 Structures of common organic impurities found in polycarbonate raw materials. (I) 4-isopropenylphenol (IPP); (II) 2′,4″-dihydroxy-2,2-diphenylpropane (o,p-BPA); (III) 2,4-bis(4-hydroxyphenyl)-4-methyl-1-penten (dimer); (IV) 2,4-bis(4-hydroxycumyl)phenol (trisphenol-I); (V) 4-(4′-hydroxyphenyl)2,2,4-trimethylchroman (chroman); (VI) 1,1-spirobis(3,3-dimethylindanol-5) (SBI); (VII) 2,4-bis(α, α-dimethyl-4-hydroxybenzyl)phenol (trisphenol-II); (VIII) 9,9-dimethylxanthene (DMX); (IX) phenyl methyl carbonate (PMC); (X) methoxyphenyl benzoate (MOB).

Concentrated ether solution of the intermediate was added to phenol in excess, dissolved in toluene, acidified with 77% H_2SO_4 and intensely stirred for 6 h. The organic solution was evaporated to dryness and the residue was separated by flash chromatography into two fractions containing mainly 2′-hydroxy-2,4,4-trimethylflavan and o,*p*-BPA. The latter was crystallized several times from isooctane and *n*-heptane.

2,4-bis(4-hydroxyphenyl)-4-methyl-1-penten (IPP-dimer)

IPP-dimer was obtained by dimerizing IPP in the presence of a strongly acidic ion-exchange resin type Amberlyst (Rohm and Haas Co., USA) in a modification of the method described by Shafer.[9] The reacting mixture was evaporated to dryness and the residue was separated by flash chromatography into three fractions. The first one (IPP-dimer) was crystallized several times from isooctane and *n*-heptane.

4-(4′-hydroxyphenyl)2,2,4-trimethylchroman (chroman)

Also known as Dianin's compound, chroman was prepared from the melted mixture of mesityl oxide and phenol. The mixture, containing the excess of phenol was saturated with dry hydrogen chloride and left for 48 h at room temperature. The crude product was collected by filtration and crystallized several times from benzene.

1,1-spirobis(3,3-dimethylindanol-5) (SBI)

SBI was prepared by a method described by Baker and Besly.[10] A solution of BPA in acetic acid and concentrated hydrochloric acid was heated on a water-bath for 48 h. After standing overnight a white, crystalline product was collected, washed with 50% acetic acid, then with water, and dried. The condensation product obtained as above was reacetylated with acetic anhydride and then crystallized three times from acetic acid. This product was sublimated over anhydrous potassium carbonate at 20 mm pressure and obtained in thick plates. Finally, SBI was obtained from its acetyl derivative by hydrolysis.

2,4-bis(4-hydroxycumyl)phenol (BPX-I) and 2,4-bis(α, α-dimethyl-4-hydroxybenzyl)phenol (BPX-II)

These two trisphenols, also identified as trisphenol-I (or BPX-I) and trisphenol-II (or BPX-II), respectively, were synthesized again in a modification of the method described by Shafer.[9] BPX-I was obtained by reacting 4-isopropylphenol with BPA whereas BPX-II was prepared by reacting 4-isopropylphenol with chroman. In both cases, reactions were conducted in the presence of an Amberlyst-15C catalyst (Rohm and Haas Co.). The reacting mixtures were evaporated to dryness and the residue was separated by flash chromatography into two fractions. In both cases, the products were crystallized several times from benzene.

9,9-dimethylxanthene

9,9-Dimethylxanthene, also known as DMX, was extracted from the brown tarry residue obtained after crude BPA melt crystallization. Residuum was dissolved in benzene, and extracted several times with 10% NaOH. The solution was evaporated and the residue vacuum distilled.

Metallic impurities

Metallic impurities were spiked as metal phenoxides. Therefore, titanium (IV) tetraphenoxide (TTTP) was prepared by reacting titanium isopropoxide and phenol (1 : 4 molar ratio) in dry toluene as in the method described by Takashi.[11] Iron (III) phenoxide, copper (II) phenoxide, and nickel (II) phenoxide were prepared by reacting corresponding metal hydroxides in aqueous solution with phenol.

All reagents used in the preparations were form Sigma-Aldrich (Tres Cantos, Madrid).

Analytical methods

Impurities determination

Organic impurities in BPA raw material were analyzed by high performance liquid chromatography in an adaptation of the method described by Poskrobko et al.,[12] using a Waters 2695 liquid chromatograph equipped with a diode array detector system Waters 2996 (Waters, Milford, MA). The analysis were performed on an X-Terra C18 column (i.d. = 150 × 2.1 mm) with methanol (phase A) and deionized water (phase B) as the mobile phase. The detector measured the UV-visible spectra at a three different wavelengths of 254, 280, and 300 nm. 2,6-xylenol was used as internal standard.

Organic impurities in DPC raw material were analyzed by gas chromatography using in an adaptation of the method described by Xing et al.[13] using an Agilent 6890*N* series gas chromatograph (Agilent, Palo Alto, CA) and a FID detector. An Agilent HP-5 capillary column (30 m × 0.25 mm id, 0.25 mm film thickness) was used for separation. Helium was used as a carrier gas at a flow rate of 1.0 mL min^{-1} with a 1 : 50 split ratio. Injection temperature was 523 K and the oven temperature was programmed from a starting value of 363 K maintained for 1 min and then increased till 503 K at a 15 K min^{-1} rate. Sample baseline chromatograms for typical BPA and DPC samples are presented in Figure 2.

Metals were determined in an external laboratory (SAIT, Murcia, Spain) by induced coupled plasma (ICP) atomic absorption.

Yellowness index

Solution YI was measured with a HACH DR/4000 UV-Vis spectrophotometer (Hach Company, Loveland, CO) provided with a temperature control module in the cell holder. Samples of 1 g. (approx.) were dissolved in 5 mL methylene chloride (Sigma-Aldrich, Tres Cantos, Madrid), then 0.45 μm membrane filtered and filled in a 1 cm quartz sample cell with a special stopper (no head space) to prevent losses from volatile solvent. For same reason, temperature was controlled to 15°C set-point. Transmittance was measured under CIE Illuminant C and the CIE 1931 2° observer conditions by scanning in the 380 to 780 nm range against a blank methylene chloride solution. Absorption curves obtained were converted to tristimulus values using standard ASTM methods D1925 and E308, respectively.[14,15] Tristimulus values are in turn converted into YI by the following equation:

$$\text{YI} = \frac{(1.2750X_c - 1.0584Z_c)100}{Y_c}$$

where X_c, Y_c, Z_c are the tristimulus values.

EXPERIMENTAL DESIGN

A two-level factorial design was used for the settings of our experiments with a total of 15 impurities as experimental factors. A full factorial design would have been unfeasible (2^{15} = 32,768 runs); therefore, a fractional factorial design was selected instead. Fractional factorial designs are very useful in factor screening because they reduce down the number of runs to a manageable size. The runs that are performed are a selected subset (fraction) of the

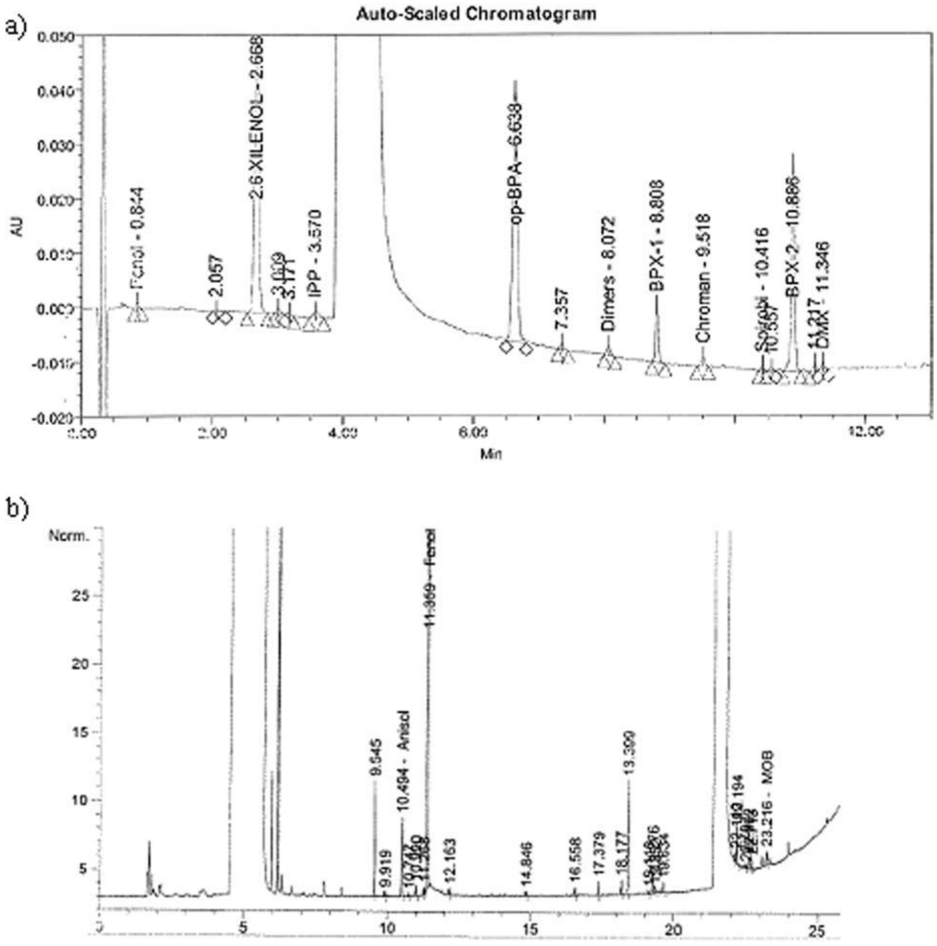


Figure 2 Raw materials impurities baseline. (a) BPA HPLC chromatogram. (b) DPC GC-MS chromatogram.

full factorial design. The main problem when not all factor-level combinations are run is that some of the effects will be confounded. Confounded effects cannot be estimated separately and are said to be aliased. Because some effects are confounded and cannot be separated from other effects, the fraction must be carefully chosen to achieve meaningful results. Choosing the "best fraction" often requires specialized knowledge of the process under investigation.

In our case, a fraction 1/512 (resolution IV) with three replicates (corner points) and a total of 193 experimental runs was selected. This fraction allows a clear determination of main effects and second order interactions for each impurity without confounding them with higher order interactions. From the nature of this phenomenon, interactions of orders 3 and above are very rare for which they can be safely ignored during the fractionation process. Given the fact that the equipment can host up to 50 simultaneous reactions, the complete experimental run could be executed in four working days. Maximum and minimum values for the levels in each factor were selected based on standard operating conditions and typical values under upsetting conditions that would lead to an unacceptable pp-BPA purity (95%) for a polycarbonate resin. Table II summarizes the experimental space.

TABLE II
Maximum and Minimum Values used in the Experimental Design

Impurity	Low value (ppm)	High value (ppm)
Isopropenyl phenol (IPP)	20	80
o,p-Bisphenol A	1000	4000
IPP-dimer	35	140
Trisphenol-I	160	640
Chroman	40	160
Spirobiindane (SBI)	15	60
Trisphenol-II	805	3220
9,9-Dimethylxanthene	45	180
Phenyl methyl carbonate (PMC)	25	100
2-Methoxyphenyl benzoate (MOB)	40	160
Anisol	20	80
Iron, ppm	50	200
Titanium, ppm	125	500
Copper, ppm	50	200
Nickel, ppm	30	120

TABLE III
Estimated Effects and Coefficients for Yellowness Index

Term	Effect	Coef	T	P-value
Constant		48.8737	313.36	0.000
opBPA	0.1307	0.0654	0.42	0.676
IPP	14.8977	7.4488	47.76	0.000
IPP-dimer	0.2945	0.1472	0.94	0.347
Chroman	−0.251	−0.1255	−0.8	0.422
SBI	−0.0888	−0.0444	−0.28	0.776
BPX-I	0.3332	0.1666	1.07	0.287
BPX-II	0.2327	0.1164	0.75	0.457
DMX	5.032	2.516	16.13	0.000
PMC	0.6017	0.3008	1.93	0.056
Anisol	0.3823	0.1911	1.23	0.222
MOB	7.32	3.66	23.47	0.000
Ti	−0.0107	−0.0054	−0.03	0.973
Cu	0.6222	0.3111	1.99	0.048
Fe	0.0555	0.0278	0.18	0.859
Ni	0.4511	0.2256	1.45	0.150
opBPA*IPP	0.0347	0.0173	0.11	0.912
opBPA*IPP-dimer	0.0724	0.0362	0.23	0.817
opBPA*chroman	−0.1141	−0.057	−0.37	0.715
opBPA*SBI	0.045	0.0225	0.14	0.885
opBPA*BPX-I	−0.2063	−0.1032	−0.66	0.509
opBPA*BPX-II	−0.3469	−0.1735	−1.11	0.268
opBPA*DMX	−0.2223	−0.1111	−0.71	0.477
opBPA*PMC	−0.3356	−0.1678	−1.08	0.284
opBPA*Anisol	0.398	0.199	1.28	0.204
opBPA*MOB	0.3167	0.1584	1.02	0.312
opBPA*Ti	−0.1557	−0.0779	−0.5	0.618
opBPA*Cu	0.112	0.056	0.36	0.720
opBPA*Fe	0.0956	0.0478	0.31	0.760
opBPA*Ni	−0.3532	−0.1766	−1.13	0.259
IPP*MOB	0.0807	0.0403	0.26	0.796
IPP*Ti	0.2058	0.1029	0.66	0.510
IPP*Cu	−0.2666	−0.1333	−0.85	0.394
IPP*Fe	−0.1824	−0.0912	−0.58	0.560
IPP*Ni	0.285	0.1425	0.91	0.362
IPP-dimer*Fe	0.4583	0.2292	1.47	0.144
IPP-dimer*Ni	−0.3822	−0.1911	−1.23	0.222
SBI*BPX-I	0.0979	0.049	0.31	0.754
SBI*Anisol	−0.1753	−0.0876	−0.56	0.575
SBI*Cu	0.0061	0.003	0.02	0.984
SBI*Ni	0.0402	0.0201	0.13	0.898
BPX-II*Fe	−0.0913	−0.0457	−0.29	0.770
BPX-II*Ni	0.0402	0.0201	0.13	0.898
PMC*Cu	−0.1596	−0.0798	−0.51	0.610
PMC*Ni	−0.1347	−0.0674	−0.43	0.666
Ti*Ni	0.1049	0.0524	0.34	0.737
Center point		0.1263	0.06	0.954

Each reactor was weighed and charged with 5 g of BPA and 4.93 g of DPC (1.05 molar ratio). Then the appropriate amount of each impurity was loaded with the help of a micropipette. Once the reactors were loaded with raw materials and impurities and before applying the temperature program, they were subjected to several cycles of nitrogen filling and vacuum extraction with the purpose of removing any oxygen trace from the reactor. Next, the contents were melted in the digester at 180°C for 30 min. As soon as the melt reached the desired experimental temperature, 100 ppb of NaOH in methanol were added to act as a catalyst. This moment was designated as the reaction start-up. The polymerization conditions were taken as follow: Stage 1 : 240°C, 1.5 h; stage 2 : 310°C, 1.5 h. Once the reaction was completed, 2 mL samples were taken and quenched in 10 mL of methylene chloride solution acidified with 100 ppb of *n*-butyl-*p*-toluenesulphonate. Finally, tristimulus values and YI were measured on this solution.

RESULTS AND DISCUSSION

Results of solution YI for all experimental runs were analysed using MINITAB^{TM} (Minitab Inc., State College, PA) to determine the statistical significance for each one of the factors. Results of the statistical model including all main effects and second order interactions are shown in Table III. In that table, factors and interactions with P-values greater than 0.05 mean that they are not statistically significant. Consequently, only three factors (IPP, DMX, and MOB) showed up as strongly statistically significant with regards to its influence on YI. Same information is displayed graphically as main effects an interaction plots in Figures 3 and 4, respectively. Only these three impurities increase YI when they are present at high concentrations as indicated by their positive coefficients. Table IV shows the ANOVA table corresponding to the fractional factorial analysis which indicates that interaction and curvature effects are negligible as established by their extremely high P-values. Figure 5 illustrate the goodness of the analysis since the residuals are normally distributed [Fig. 5(a)] and show a totally random appearance meaning that no distortion is introduced by the execution order [Fig. 5(b)].

Finally, a multiple regression analysis was conducted on the YI data set including only IPP, DMX, and MOB as parameters. Table V shows the ANOVA table corresponding to this multiple regression analysis which fitted the following expression:

$$\mathrm{YI} = 26.2 + 0.248\ \mathrm{IPP} + 0.0373\ \mathrm{DMX} + 0.0610\ \mathrm{MOB}$$

The regression coefficient indicates that 94.6% of the residual sum of squares can be explained by the regression model letting only a 5.4% to experimental error or other minor factors. Given the fact that the experimental design was quite large, and replicates were provided only for the corners points, this is statistically a good fit.

The fact that DMX arises as a significant factor is not surprising. Derivatives of 10H-9-oxaanthracene are commonly referred to collectively as xanthenes, and among other uses are the basis of a class of dyes which includes fluorescein, eosins, and rhodamines. On the other hand, MOB can rearrange into color bodies with structures similar to xanthones.

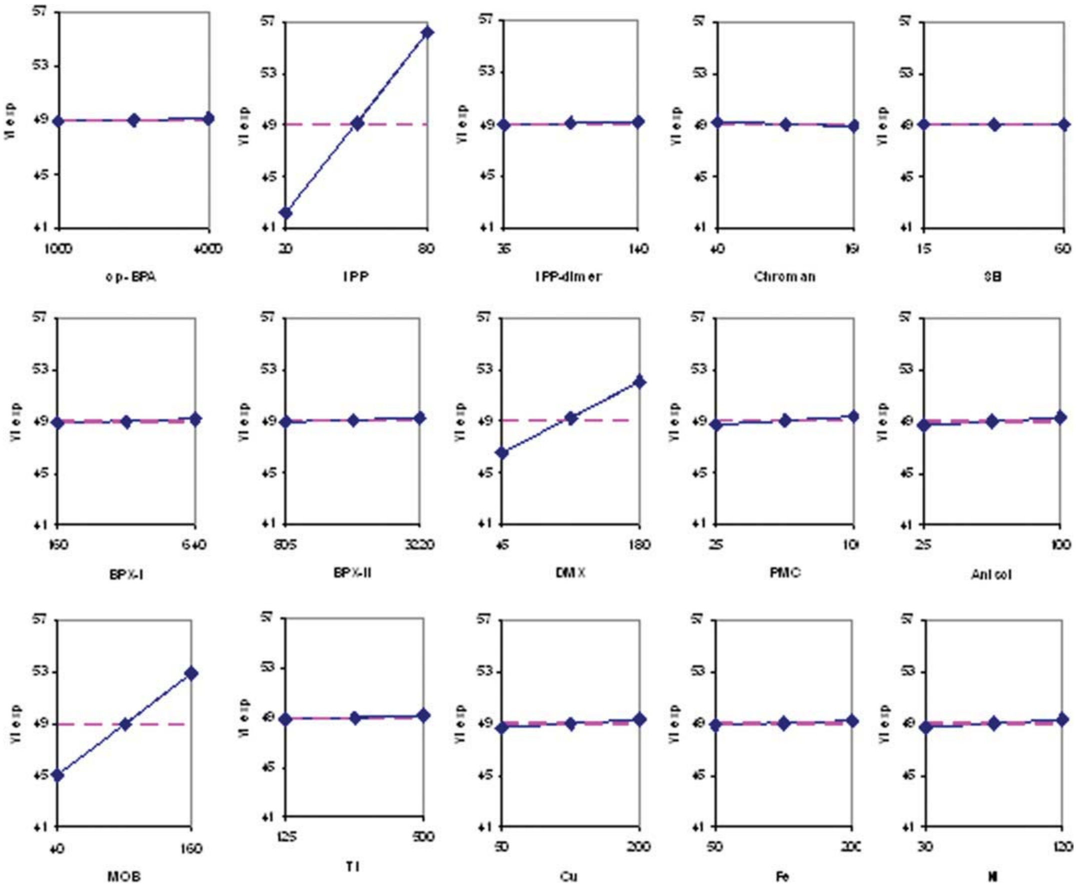


Figure 3 Main effects plot. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

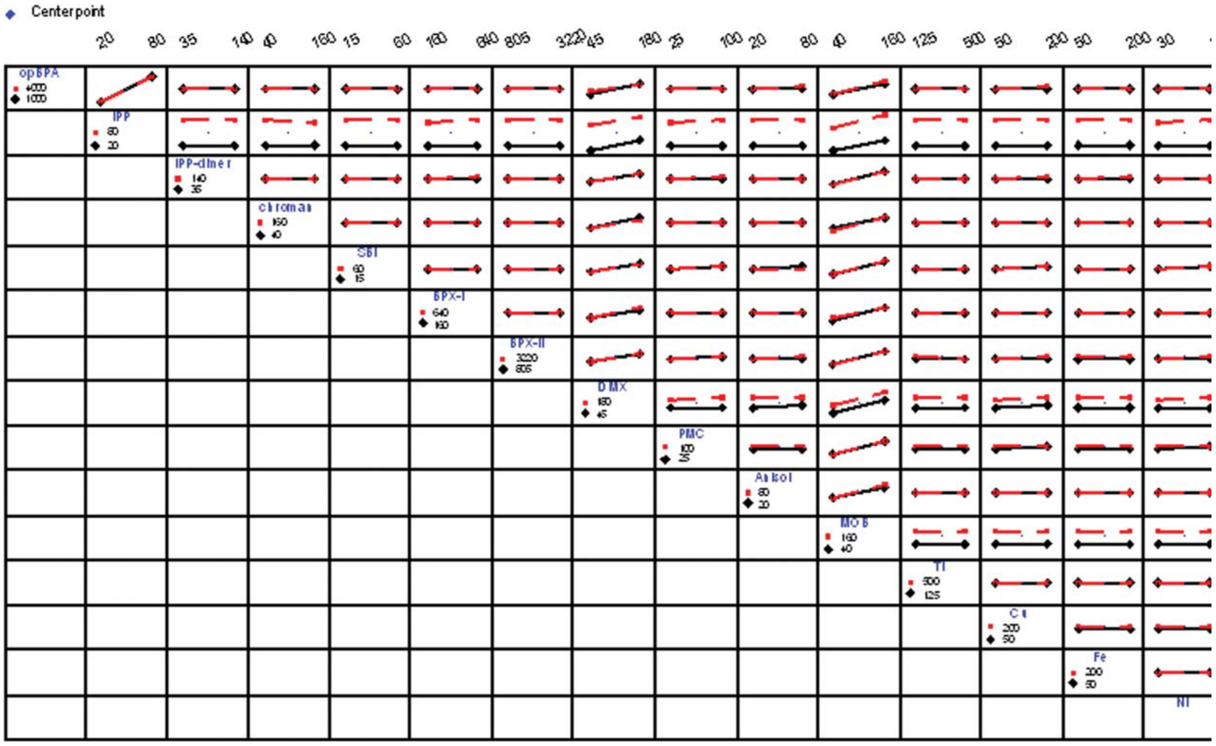
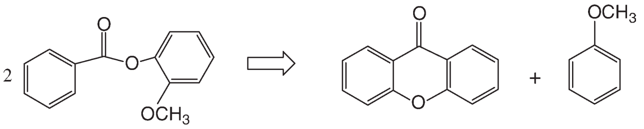


Figure 4 Interactions plot. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

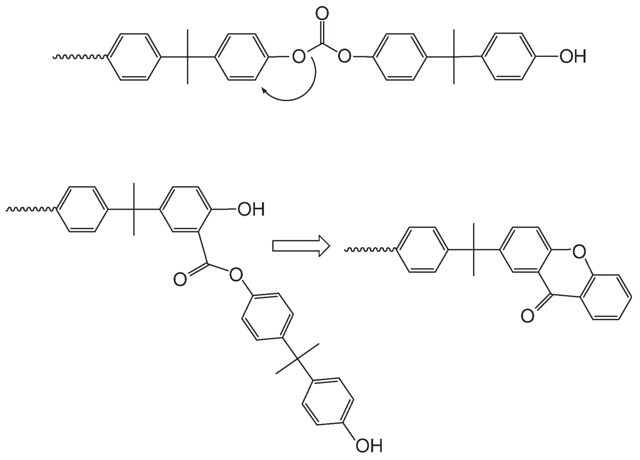
TABLE IV
ANOVA Table for the Fractional Factorial Analysis Design

Source	DF	Seq SS	Adj SS	Adj. MS	*F*	*P*
Main effects	15	14509.7	14509.7	967.311	207.1	0
2-Way Interactions	30	70.7	70.7	2.357	0.5	0.985
Curvature	1	0	0	0.016	0	0.954
Residual error	146	681.9	681.9	4.671		

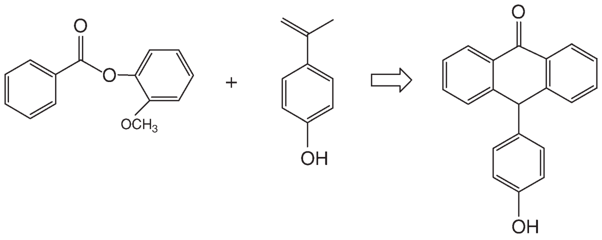
Xanthones are an organic kind of yellow pigments found in a variety of tropical trees such as mangosteen tree (*Garcinia mangostana*) and as a constituent of plants in the families *Bonnetiaceae*, *Clusiaceae* and in some species in the family *Podostemaceae*. Under heating and catalyst, xanthones can be produced by the heating of phenyl salicylate and their derivatives.[16] MOB can rearrange in a similar way to produce:



This can be related with the observations made by others that polycarbonate resins with a high content of Fries product also show YI values higher than normal.[17] Fries rearrangement in polymer chains can give also xanthone type structures:



IPP and MOB also can give condensation products to form xanthene type structures:



Xanthones and xanthenes are well known for being dyes in the yellow range of the chromatic scale.

Another theory that could be consistent with the experimental data is that oligomer structures containing IPP, MOB, and DMX could undergo a free radical process (caramelization) leading to color bodies similar to those obtained in the thermal treatment of saccharine. These processes take place at

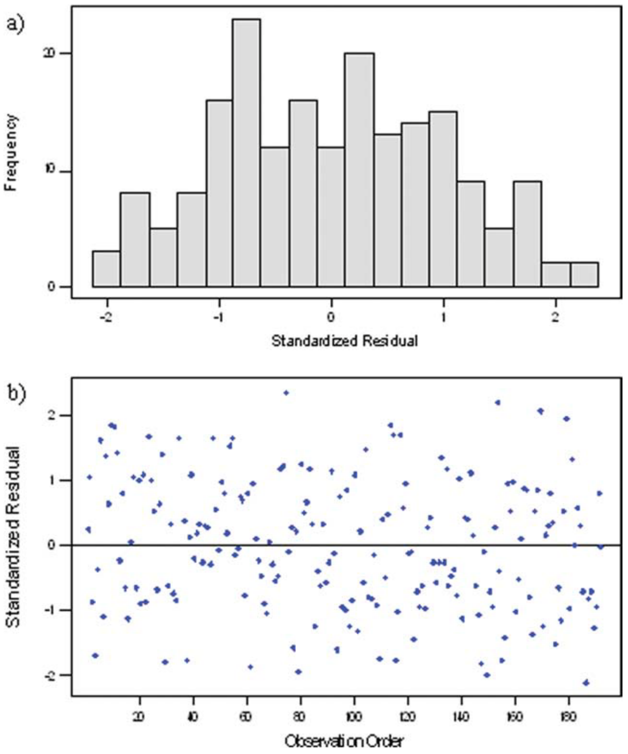


Figure 5 Statistical validation. (a) Histogram of residuals. (b) Residuals vs. the order of the data. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TABLE V
ANOVA Table for the Regression Analysis

Predictor	Coef	SE Coef	*T*	*P*	
Constant	26.1663	0.4596	56.93	0.000	
IPP	0.248294	0.005016	49.5	0.000	
DMX	0.037274	0.00223	16.72	0.000	
MOB	0.061	0.002508	24.32	0.000	
S =	2.085				
R-Sq =	94.60%				
R-Sq(adj) =	94.50%				
Source	DF	SS	MS	*F*	*P*
Regression	3	14,440.5	4813.5	1106.97	0.000
Residual error	189	821.8	4.3		
Total	192	15,262.3			

similar temperatures as those found in the polymerization process and require the presence of a basic catalyst.[18]

CONCLUSIONS

The main purpose of this work was to study the influence of raw materials impurities on polycarbonate color. Main contributors to color are found in the organic fraction contained in the raw materials, being IPP, MOB, and DMX. Contribution of metallic impurities is negligible. A quantitative expression which predicts YI upsets under certain concentrations of these impurities has been found. This correlation can be used to predict potential manufacturing issues or in establishing tighter specifications for raw materials suppliers.

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