Concentrations of bisphenol A in the composite food samples from the 2008 Canadian total diet study in Quebec City and dietary intake estimates

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A total of 154 food composite samples from the 2008 total diet study in Quebec City were analysed for bisphenol A (BPA), and BPA was detected in less than half (36%, or 55 samples) of the samples tested. High concentrations of BPA were found mostly in the composite samples containing canned foods, with the highest BPA level being observed in canned fish (106 ng g⁻¹), followed by canned corn (83.7 ng g⁻¹), canned soups (22.2–44.4 ng g⁻¹), canned baked beans (23.5 ng g⁻¹), canned peas (16.8 ng g⁻¹), canned evaporated milk (15.3 ng g⁻¹), and canned luncheon meats (10.5 ng g⁻¹). BPA levels in baby food composite samples were low, with 2.75 ng g⁻¹ in canned baby foods. BPA was also detected in some foods that are not canned or in jars, such as yeast (8.52 ng g⁻¹), baking powder (0.64 ng g⁻¹), some cheeses (0.68–2.24 ng g⁻¹), breads and some cereals (0.40–1.73 ng g⁻¹), and fast foods (1.1–10.9 ng g⁻¹). Dietary intakes of BPA were low for all age–sex groups, with 0.17–0.33 µg kg⁻¹ body weight day⁻¹ for infants, 0.082–0.23 µg kg⁻¹ body weight day⁻¹ for children aged from 1 to 19 years, and 0.052–0.081 µg kg⁻¹ body weight day⁻¹ for adults, well below the established regulatory limits. BPA intakes from 19 of the 55 samples account for more than 95% of the total dietary intakes, and most of the 19 samples were either canned or in jars. Intakes of BPA from non-canned foods are low.

Keywords: total diet; gas chromatography (GC); gas chromatography/mass spectrometry (GC/MS); total diet studies bisphenol A

Introduction

Bisphenol A (BPA) is used as a monomer in the production of polycarbonate plastics and epoxy resins, and as an additive for the elimination of surplus hydrochloric acid in the production of polycarbonate (PVC) organosols. Polycarbonate is used in food storage containers such as water bottles and baby bottles, while epoxy resins and PVC organosols are used in the internal coating for food and beverage cans, and also in the internal coating on metal lids for foods in glass jars to protect the food from direct contact with metal. Residues of BPA in polycarbonate plastic containers and coatings can migrate into foods, especially at elevated temperatures, thus humans are inevitably exposed to BPA primarily through the diet. Since BPA is an endocrine disruptor that mimics the action of the hormone oestrogen, the specific migration limit for BPA in food or food simulants was set at 0.6 µg g⁻¹ by the European Commission Directive in an amending document relating to plastic materials and articles intended to come into contact with foodstuffs (European Commission 2004). The maximum acceptable dose for BPA was established at 50 µg kg⁻¹ body weight day⁻¹ by the US Environmental Protection Agency (USEPA) (1993) and the tolerable daily intake (TDI) of 50 µg kg⁻¹ body weight day⁻¹ established by the European Food Safety Authority (EFSA) (2006). The provisional tolerable daily intake for BPA established by Health Canada (2008) was 25 µg kg⁻¹ body weight day⁻¹.

Levels of BPA in various canned food products have been determined in targeted surveys from several countries (Yoshida et al. 2001; Goodson et al. 2002; Thomson and Grounds 2005; Sajiki et al. 2007; Yonekubo et al. 2008; Cao et al. 2008, 2009a, 2009b), and these results were used for the BPA dietary exposure assessments (Government of Canada 2008). The recent dietary exposure assessment of pregnant women to BPA conducted in Spain (Mariscal-Arcas et al. 2009) and the risk assessment of BPA for adults in Korea (Lim et al. 2009) were also based on canned...
foods. BPA is rarely measured in non-canned foods, however. Thus, the contribution from the non-canned foods to the overall dietary intake of BPA is unknown. Information is also limited on the effect of food processing, preparation and cooking procedures on BPA levels in the final cooked foods. Since polycarbonate tools and containers and containers with epoxy coatings may be used during food preparation for cooking, BPA could be introduced into the final cooked foods due to migration from polycarbonate and coatings.

Dietary exposure to chemical contaminants can be better estimated using the results from total diet studies than from the individual raw foods since the food samples are processed as for consumption and thus any increase or decrease of the chemicals during processing is included. A total diet study consists of purchasing foods commonly consumed, processing them as for consumption, combining the foods into food composites, homogenizing them, and analysing them for chemical contaminants. The design of total diet studies covers most foods consumed, will include food groups that may otherwise be missed in surveys targeted at canned foods only, and a thus total diet study is a good way of determining average population exposure across the diet and is recommended by the World Health Organization (WHO) (2005) as the most cost-effective way of determining average population exposure across the diet and is recommended by the World Health Organization (WHO) (2005) as the most cost-effective approach to assess actual dietary intakes of both toxic and nutritionally important chemicals. On the other hand, the disadvantage of a total diet study is that foods are composited and thus the levels of contaminant in individual foods and the variability across brands are lost. The Canadian total diet study has been ongoing since 1969 to monitor various chemical contaminants in the Canadian food supply and plays an important role in generating data for human exposure assessments. In this study, total diet food composite samples were analysed for BPA for the first time ever, and the results were used to estimate the dietary intakes of BPA for different age and sex groups of Canadian populations.

**Materials and methods**

**Materials and reagents**

Acetonitrile (HPLC grade) and methanol (HPLC grade) were purchased from J.T. Baker (Phillipsburg, NJ, USA). Bisphenol A-d16 (>99% D), toluene (glass distilled), potassium carbonate (ACS grade), bisphenol A (99%), isoctane (pesticide-residue grade), MTBE (methyl t-butyl ether, 99.9%), K2HPO4 (ACS), Na2SO4 (anhydrous, ACS grade), 1-pentanol (99%) and dodecane (99%) were purchased from Sigma-Aldrich (Oakville, ON, Canada). Acetic anhydride (ACS grade) and H3PO4 (85%, HPLC grade) were purchased from Fisher (Ottawa, ON, Canada).

BPA and BPA-d16 standard solutions were prepared in acetonitrile and stored at 4°C. The pH 7 phosphate buffer was prepared by dissolving 28.6 g of Na2HPO4 in 2 L of de-ionized water; and the pH was adjusted to 7.0 ± 0.1 with H3PO4. The 1.0 M K2CO3 solution was prepared by dissolving 69 g of anhydrous K2CO3 in 500 ml of de-ionized H2O. Derivatized BPA calibration standard solutions (10–200 ng ml⁻¹) were prepared by adding BPA standard solution to 22 ml vials containing 10 ml of 1.0 M K2CO3 solution, and by going through the derivatization procedure together with the samples.

**Sample collection**

Foods from four different stores in Quebec City were collected over a 5-week period starting in September 2008. The foods were prepared as for consumption (Conacher et al. 1989) by the Department of Food Science, Kemptville College, University of Guelph, and combined into food composites according to the established procedures for each one of the composites. Composites for foods that can be consumed both raw and cooked (e.g., cauliflower, carrots, broccoli, tomatoes, spinach) were prepared as a mixture of the raw and cooked (1:1). Composites for foods that are available in different types of containers (e.g., beer in glass bottle and can) were also prepared as a mixture (1:1). Stainless steel or glass vessels were used for all processing. Drinking water from Kemptville College was used for food processing. Food composites were stored frozen at −20°C until analysis.

**Sample extraction, derivatization and analysis**

Details of the method for sample extraction, derivatization and analysis can be found elsewhere (Cao et al. 2008, 2009a, 2009b). Briefly, approximately 2–6 g of sample were weighed into a 15-ml polypropylene centrifuge tube. The sample was spiked with internal standard BPA-d16 and extracted with acetonitrile. The extract was diluted with pH 7.0 phosphate buffer solution, purified by going through the C18 solid-phase extraction (SPE) cartridge, and eluted with 50% acetonitrile/water. The extract was concentrated and derivatized to the di-ester using acetic anhydride in a K2CO3 solution. The extract was derivatized in water, and the di-ester derivative was extracted with isoctane followed by methyl t-butyl ether and analysed using an Agilent 6890 gas chromatograph (GC) coupled to a 5975 mass selective detector (MSD) in selected ion-monitoring mode (ions m/z 213, 228, 270, 312, for BPA and 224 for BPA-d16). Isotope dilution was used to determine BPA levels in samples. Each batch of analysis included two method blanks (de-ionized water), two duplicate samples (i.e., two composite samples). Isotope dilution was used to determine BPA levels in samples. Each batch of analysis included two method blanks (de-ionized water), two duplicate samples (i.e., two composite samples).
samples extracted and analysed twice), two samples spiked with BPA, and a blank (water) spiked with BPA. Concentrations are expressed on a wet weight basis throughout the paper. The average difference between the results from the duplicate samples was 20%. The method was validated for various food samples previously (Cao et al. 2008, 2009a, 2009b); the estimated method detection limits based on ten times the signal-to-noise ratio were 0.14 ng g$^{-1}$ for simple liquids such as water and beverage, 0.20 ng g$^{-1}$ for some dairy products and other liquids, 0.38 ng g$^{-1}$ for simple food matrices such as fruits and vegetables, and 1.0 ng g$^{-1}$ for complex food matrices such as fast foods.

Dietary intake estimates
Dietary intakes of BPA for different age–sex groups of children and adults were calculated by multiplying the concentration of BPA in a food composite by the amount of this food product consumed by each age–sex group based on the 24-h diet recall from the Nutrition Canada Survey (Health Canada 1977; Conacher et al. 1989; Dabeka et al. 1993). The total BPA intake was then calculated by summing the intakes from all food composites, and dividing by the average body weight of each age–sex group (Health Canada 1977). For the intake calculations, the average of the two duplicates (if available) were used, while zero was used for samples where BPA was not detected.

Results and discussion
BPA concentrations
The food composite samples from the Canadian total diet study include a variety of food categories including dairy products, meat and meat products, poultry and fish, cereals and cereal products, vegetable and vegetable products, fruit and fruit products, baby foods, fast food, soups, beverages, and other miscellaneous foods (e.g., candy). The list of food composite samples is revised from time to time to reflect the changes in the diet of the average Canadian population. In the 2008 Canadian total diet study conducted in Quebec City, a total of 154 food composite samples were collected and analysed for BPA. Among the 154 composite food samples, BPA was detected in only 55 composite samples (Table 1) with concentrations ranging from 0.20 to 106 ng g$^{-1}$, well below the specific migration limit of 0.6 μg g$^{-1}$ for BPA in food and food simulant established by a European Commission (2004) Directive. However, it should be mentioned that since the composite sample is a mixture of four different individual samples obtained from different stores, BPA levels in some of the individual samples could be much higher than the composite. BPA was not detected in the other 99 composite food samples (Table 2).

BPA was not detected in majority of the dairy products. The highest BPA level, 15.3 ng g$^{-1}$, was detected in the canned evaporated milk sample, which is expected to be due to migration from the can coating. BPA was also detected in two cheese samples at 2.24 and 0.68 ng g$^{-1}$. The exact sources for BPA in the cheese samples are not known. Migration from the packaging paper, especially the plastic packaging film, is a possibility since it is known that BPA is used as an additive for elimination of surplus hydrochloric acid in the production of PVC products, and it is also reported that BPA is used as an additive in food packaging PVC films (Lopez-Cervantes and Paseiro-Losada 2003). BPA could also be introduced during the production process if equipments/containers have epoxy coatings.

BPA was detected in the canned luncheon meat composite sample at 10.5 ng g$^{-1}$; migration from the can coating is very likely the source. BPA levels in shellfish, marine and fresh water fish samples were low. However, BPA was detected at 106 ng g$^{-1}$ in the canned fish composite sample. BPA was detected in all three canned soup composite samples, with levels ranging from 22.2 to 44.4 ng g$^{-1}$. It was not detected in the dehydrated soup sample.

Low levels of BPA (0.4–1.73 ng g$^{-1}$) were observed in a few bread and cereal composite samples. Since BPA was also observed in baking powder (0.64 ng g$^{-1}$) and especially yeast (8.52 ng g$^{-1}$), the low background BPA levels found in bread and related samples could be due to the presence of BPA in the yeast and baking powder used. In comparison with the level of BPA detected in baking powder, the exact reasons for the higher BPA level in the yeast composite sample are not known.

Although the yeast samples purchased were contained in jars with metal lids, the coating on the metal lids is unlikely to be the source of BPA in the yeast since the yeast is not in contact with the lids and migration of BPA from coating into solids at room temperature is negligible. BPA in the yeast sample is more likely introduced during the production stage instead.

BPA levels in the raw vegetable samples (non-canned) were low, while BPA was detected in all vegetable composite samples where canned products were used in part or exclusively for the preparation of composite samples. BPA was detected in the corn composite sample at 83.7 ng g$^{-1}$. Since both frozen and canned corns were used to prepare the composite, BPA level in the canned corn sample may have been higher than 83.7 ng g$^{-1}$. The canned baked bean sample had the next highest level of BPA at 23.5 ng g$^{-1}$, followed by frozen and canned peas (16.8 ng g$^{-1}$), raw and canned string beans (5.59 ng g$^{-1}$), and raw and canned beets (3.45 ng g$^{-1}$). BPA was detected in both the canned vegetable juice and the canned tomatoes and tomato sauce samples, but the levels were low, 0.53 ng g$^{-1}$ and 2.59 ng g$^{-1}$, respectively.
Levels of BPA in fruit and fruit products were low; BPA was not detected in most of the samples, even for some canned samples such as applesauce and peaches. BPA concentration in canned cherries was 3.24 ng g\(^{-1}\), followed by 1.2 ng g\(^{-1}\) in the canned pineapple sample.

BPA was not detected in any of the bottled water samples, which agrees with the results from the recent survey on BPA in bottled water products (Cao and Corriveau 2008). BPA was not detected in the water used in preparation of some of the total diet samples in this study. BPA levels in fruit juices and other beverages were also very low, below detection limits in most cases. BPA was observed in bottled wine sample at level of 0.74 ng g\(^{-1}\), possibly due to contamination during the production stage. Levels of BPA in canned beer and soft drink samples were low.

BPA concentration in the canned liquid infant formula composite sample was 2.75 ng g\(^{-1}\) (Table 1), very close to the lowest BPA levels in canned liquid infant formula products in the recent Canadian survey of infant formulae (Cao et al. 2008). However, BPA was not detected in the powdered infant formula composite sample.

BPA was detected in most of the jarred baby food composite samples. The levels observed were low (0.84–2.46 ng g\(^{-1}\)) and within the range reported in baby food products from a recent survey (Cao et al. 2009b). The low BPA levels in the baby foods in jars may be due to epoxy coatings and PVC gaskets on the metal lids, although the processing equipment and storage containers with epoxy coating and/or plastic parts also could be possible sources of BPA.

BPA was detected in most of the fast food composite samples, but the levels were low in general except for the hamburger (10.9 ng g\(^{-1}\)). The relatively high BPA levels in the hamburger may be due to the wrapping paper, although BPA may have already been in the ingredients (ground beef, cheese, sauce, bread etc.) used to make the hamburger. BPA was detected in only five of the miscellaneous food composite samples, with 0.64 ng g\(^{-1}\) in baking powder and as high as 8.52 ng g\(^{-1}\) in the yeast samples.

### Dietary intakes

Dietary intakes of BPA were calculated for different age–sex groups and are shown in Table 3. They are all well below the provisional tolerable daily intake (about one-tenth or less of 25 µg kg\(^{-1}\) body weight day\(^{-1}\)) established by Health Canada. Although the absolute dietary intakes of BPA for children aged 1 year old and up (3.3–4.7 µg day\(^{-1}\)), and for adults (3.3–5.8 µg day\(^{-1}\)), were higher than those for the infants (1.2–2.2 µg day\(^{-1}\)), dietary intakes of BPA calculated on a per kg body weight basis for the infants (0.22–0.33 µg kg\(^{-1}\) body weight day\(^{-1}\)) were much higher than those for the adults (0.052–0.081 µg kg\(^{-1}\) body weight day\(^{-1}\)).
The dietary intakes of BPA for the infants calculated from this study (0.22–0.33 μg kg\(^{-1}\) body weight day\(^{-1}\)) are close to the average dietary intakes of infants calculated from our previous study (0.21–0.50 μg kg\(^{-1}\) body weight day\(^{-1}\)) (Cao et al. 2008). The dietary intakes of BPA for the adults (0.052–0.081 μg kg\(^{-1}\) body weight day\(^{-1}\)) are close to the probable daily intake for general population (0.18 μg kg\(^{-1}\) body weight day\(^{-1}\)) from food packaging uses estimated in 1995 (Health Canada 2008) and the lower end of the exposure to BPA for the general population of Canada (0.08–4.30 μg kg\(^{-1}\) body weight day\(^{-1}\)) estimated recently (Government of Canada 2008).

BPA was detected in less than half (36%, or 55 samples) of the 154 food composite samples. As can be
Table 3. Dietary intakes of BPA for different age-sex groups.

| Dietary intake | 0–1 months, M and F | 2–3 months, M and F | 4–6 months, M and F | 7–9 months, M and F | 10–12 months, M and F | 1–4 years, M and F | 5–11 years, M and F | 12–19 years, M | 20–39 years, M | 40–64 years, M | 65+ years, M | 12–19 years, F | 20–39 years, F | 40–64 years, F | 65+ years, F | All ages Canadian, M and F |
|----------------|---------------------|---------------------|---------------------|---------------------|---------------------|---------------------|---------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| µg/day         | 1.2                 | 1.5                 | 1.6                 | 1.4                 | 2.2                 | 3.3                 | 4.4                 | 4.7              | 5.8             | 5.3             | 4.2             | 4.1             | 4.1             | 4.4             | 3.3             | 4.5             |
| µg kg⁻¹ body weight day⁻¹ | 0.33              | 0.27               | 0.23               | 0.16               | 0.22               | 0.23               | 0.17               | 0.088           | 0.081           | 0.074           | 0.059           | 0.082           | 0.071           | 0.073           | 0.052           | 0.075           |

Note: F, female; M, male.
Table 4. Per cent of BPA intake from main food composites to the total dietary intakes of BPA for different age-sex groups.

| Food Composite | 0–1 | 2–3 | 4–6 | 7–9 | 10–12 | 1–4 | 5–11 | 12–19 | 20–39 | 40–64 | 65+ | All ages
|----------------|-----|-----|-----|-----|-------|-----|-----|-------|-------|-------|-----|----------|
| All ages Canadian, M and F | 33.3 | 73.1 | 32.0 | 18.5 | 2.3 | 4.7 | 2.3 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Evaporated milk, canned | 47.0 | 2.3 | 3.3 | 28.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Fish, canned | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 1.5 | 4.5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Soups, creamed, canned | 0.0 | 20.8 | 0.0 | 20.4 | 36.4 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Bread, white | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Baked beans, canned | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 3.2 | 2.1 | 5.1 | 9.0 | 9.0 | 9.0 | 9.0 |
| Corn, frozen plus canned (1:2) | 0.0 | 2.4 | 1.4 | 0.0 | 25.9 | 25.9 | 25.9 | 25.9 | 25.9 | 25.9 | 25.9 | 25.9 |
| Peas, frozen plus canned (1:2) | 0.0 | 4.9 | 1.1 | 0.0 | 2.3 | 2.3 | 2.3 | 2.3 | 2.3 | 2.3 | 2.3 | 2.3 |
| Apple juice, canned | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Wine, bottled | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Dinners, cereal plus vegetable plus meat, in jar | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Fruit, apple or peach, in jar | 0.0 | 0.6 | 3.9 | 5.3 | 5.3 | 3.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Meat, poultry or eggs, in jar | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| Vegetables, peas, in jar | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |

Note: F, female; M, male.
seen from Table 4, BPA intakes from 19 of the 55 samples account for more than 95% of the total dietary intakes, and most of the 19 samples were either canned or in jars, thus the principal source of BPA in foods is most likely from the migration from can coating. Compared with canned foods, contributions from non-canned foods to the overall BPA intakes are low. For example, white bread accounted for only 0.6% of the total dietary intakes of BPA for all ages Canadians. BPA intakes from the other 36 food composites not listed in Table 4 account for less than 5% of the total dietary intakes.

It should be mentioned that the dietary intakes of BPA were estimated based on the food intake data from the Nutrition Canada Survey in the 1970s since the recent food intake data are not yet available. Thus, some of the data in Table 4 may not reflect the current situation in BPA exposure due to the changes in consumption patterns over the past 40 years. For example, the use of canned evaporated milk products was more popular in infant nutrition than commercial infant formula in the 1950–1970s (Fomon 2001). This is reflected by the estimated 32–74% contribution to total BPA dietary intake in infants from zero to 9 months of age (Table 4). However, based on current infant feeding practices, commercial infant formula products would be expected to be a more significant source of dietary BPA intake than evaporated milk.

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