Flavor Retention in Pregelatinized and Internally Flavored Starch Extrudates[1]

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Cereal Chem. 74(4):396–399

ABSTRACT

Corn starch (25% amylose content) was pregelatinized in a twin-screw extruder. The extrudate was ground and reextruded after adjusting moisture content to 9, 13, or 17% (db) and blending with 5% (w/w) of flavor compounds cinnamaldehyde, eugenol, nonanoic acid, or 3-octanone. Initial moisture content significantly influenced radial expansion, specific mechanical energy, and flavor retention. Significantly higher flavor retention was obtained when flavor was injected into the extruder barrel as compared to preblending of flavors in pregelatinized starch. Flavor retention upon extrusion was lower with pregelatinized starch than with raw starch.

Considerable efforts have been concentrated in recent years on developing new snacks, cereal breakfast foods, pet foods, and modified starches using extrusion cooking. Extrusion cooking is presently considered a high-temperature short-time process. Extrusion processing subjects raw ingredients to high temperatures, shearing stresses, and pressure, thereby significantly changing physical, molecular, and sensory attributes of the product.

Traditional flavoring of extrudates require surface application of flavors in a tumbler together with an oil spray. This results in a weight gain of up to 25% oil (Lyon 1980). Increased awareness about nutrition and consumer preference for low calorie and low fat foods has led the snack food industry to identify alternate flavoring techniques such as internal flavor application. Several researchers studied factors influencing flavor retention in extrudates (Blanchfield and Ovenden 1974, Kinsella 1978, Palkert and Fagerson 1980 Lane 1983, Lazarus and Renz 1985, Chen et al 1986, Sadafian and Crouzet 1988, Maga 1989, Nair et al 1994, Villota and Hawkes 1994). Flashing off of volatiles (Kinsella 1978), thermal degradation, oxidation, and polymerization of added flavors during extrusion (Blanchfield and Ovenden 1974, Palkert and Fagerson 1980, Kollengode et al 1996a) were identified as the causes of flavor losses in internally flavored extrudates.

Suggested processes for enhancing flavor retention by internal application techniques include physical protection of flavors by encapsulation or by cyclodextrin complexing (Blanchfield and Ovenden 1974, Sadafian and Crouzet 1988), use of flavor precursors (Blanchfield and Ovenden 1974, Fischetti 1975) and injection of flavors (Kollengode et al 1996a).

Preliminary studies showed that pregelatinized starch could be extruded at a lower feed moisture content (9%, db) than raw starch (15%). Kollengode et al (1996a) reported higher flavor retention in extruded starch with lower feed moisture content (feed mc). No systematic study has been reported on enhancing flavor retention by using pregelatinized starch at lower moisture contents. Therefore, this study was conducted to investigate the influence of pregelatinized starch on volatiles retention in internally flavored extrudates in the 9–17% mc range. The effects of moisture content of pregelatinized starch on the radial expansion ratio and the specific mechanical energy (SME) requirements also were studied.

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Publication no. C-1997-0612-01R.

MATERIALS AND METHODS

Materials

Normal corn starch (25% amylose content) was obtained from American Maize Products (Hammond, IN). The powdered starch was agglomerated to minimize fines (which hinder feeding) by steadily spraying distilled water onto the starch in an inclined and rotating pan and drying to 9–12% mc at room temperature. Starch moisture content was adjusted to 18% before gelatinizing starch in a twin-screw extruder. The pregelatinized starch was then adjusted to 9, 13, and 17% mc for subsequent extrusions with the flavor compounds.

Selection of flavor compounds was based on molecular weight (<250 Da), boiling point (>165°C) and compound cost. Selected flavors were not inherent in either native or extruded starch. Flavors were selected to produce model systems with volatile compounds to serve as unique markers with different functional groups (alcohol -OH, aldehyde -CHO, carboxylic acid -COOH, and ketone -CO). Research-grade cinnamaldehyde, eugenol, nonanoic acid, and 3-octanone were obtained from Aldrich Chemical Co. (Milwaukee, WI). Porapak Q, which was used for headspace analyses, was obtained from Supelco, Inc. (Bellefonte, PA).

Methods

Flavors either were blended at 5% (w/w) (level determined from earlier study by Kollengode et al [1996a]) directly in a Hobbart mixer with the pregelatinized starch. Flavors were added dropwise, blended for 15 min, placed in air-tight plastic containers, equilibrated overnight at room temperature, and then extruded, or they were injected into the third port (Fig. 1) of the extruder using a piston pump (Rainin Instruments Co., Woburn, MA) during extrusion. Flavor blending was done after adjusting the moisture content of the pregelatinized starch. No solvent was used during flavor addition.

Extrusions were performed in a twin-screw laboratory extruder (model 2803, Plasticorder, C. W. Brabender Instruments, Hackensack, NJ) with a 1.9-cm barrel diameter, a 20:1 barrel length-to-diameter ratio and a 7-mm cylindrical die nozzle. Pregelatinization barrel temperatures of feed, compression, metering, and die sections were maintained at 60, 140, 140, and 140°C, respectively. Screw speed was maintained at 100 rpm. Flavored pregelatinized starch was extruded at barrel temperatures of 60, 120, 110 and 100 °C, respectively. Radial expansion ratio was calculated by dividing the cross-sectional area of the extrudate by the cross-sectional area of the die nozzle (Kollengode et al 1996b). Each value was a mean of 10 observations.

SME was determined by dividing the net mechanical energy input to the screw by the extrudate flow rates (db) (Kollengode et al 1996b). The net mechanical energy input to the extruder was calculated from torque and angular velocity measurements. Torque input to the extruder was recorded by a computer interface and controller unit (PL 2000 controller, C. W. Brabender). The angular velocity was calculated from the screw speed and diameter.

Flavor quantitation was done by the method of Kollengode et al (1996a). Preliminary tests were conducted to quantitatively extract all of the added flavors from the samples. A 1-g sample of the flavored and unextruded pregelatinized starch and a 3-g sample of extrudate were ground in an analytical mill (Cole-Parmer Instruments Co., Niles, IL) for 1 min. Simultaneously, the headspace of the mill was constantly purged with nitrogen gas (50 mL/min) for 20 min. Volatiles escaping along with N_2 were trapped using a previously conditioned Porapak-Q column (containing 0.5 g Porapak-Q with glass wool plugs on both ends). Subsequently, the ground sample was quickly transferred to a 10-mL capped glass tube to which 5 mL of HPLC grade methanol (Baxter Chemicals, McGaw Park, IL) was added. The tube was intermittently shaken (Vortex-Gene Mixer, Scientific Products, McGaw Park, IL) for 1 hr. The tube was centrifuged for 5 min at 1,215 × *g*. The supernatant (1 mL) was transferred to a capped vial. A 1-μL sample of supernatant was injected into the gas chromatograph using 2-heptanone as the internal standard. The column containing Porapak-Q was removed and saturated with HPLC-grade methanol and slowly washed with 1 mL of methanol. The elutant was collected in a capped vial. From that, 1 μL was injected into the gas chromatograph (model HP5890, series II, Hewlett-Packard Co., Avondale, PA) to determine headspace volatiles in the sample. Flavor retention was the sum of the headspace volatiles and the methanol-extracted fraction. The gas chromatograph was used with a with a capillary column (SE-30, Alltech Associates, Inc., Deerfield, IL).

Conditions of the gas chromatograph were: oven temperature 40°C for 4 min, ramp-programmed at 5°C /min to 100°C, held at 100°C for 5 min; nitrogen carrier gas at 138 kPa, split ratio 50:1; column inlet 300°C; flame-ionization detector 350°C. Peaks were integrated with a Hewlett Packard integrator. A standard curve for each flavor component was obtained by plotting known concentrations on the abscissa versus the response (area) on the ordinate. Flavor retention was calculated from the integrator response as: Flavor retention = [(sample area) × (internal area to standard ratio) /(standard area) × (internal standard area to sample ratio)] × flavor concentration in standard solution.

Statistical Analysis

Data were analyzed using a statistical computer package (SAS 1989). The experimental design was a randomized complete block design with blocking over replicates and type of flavor compound, with three replicates for each treatment.

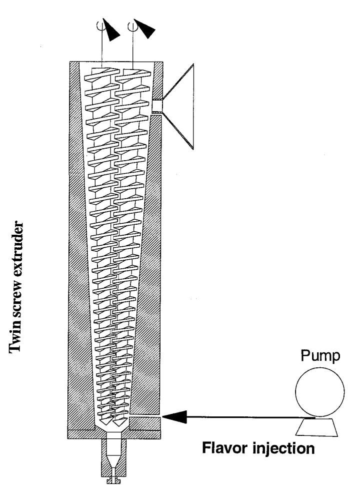

Fig. 1. Injection of flavors into the barrel of twin-screw extruder.

RESULTS AND DISCUSSION

Influence of Moisture Content and Flavor Type

The effects of feed mc (pregelatinized starch) and flavor type on radial expansion ratio are shown in Fig. 2. Feed mc had a significant ($P < 0.0035$) influence on radial expansion ratios. Radial expansion at 13% feed mc was significantly different from the 9 and 17% feed mc for all flavors except nonanoic acid. However, flavor type did not influence radial expansion ratio significantly, which was contradictory to the results obtained for extrusion of raw starch by Kim and Maga (1994). They observed that extrusion temperature and flavor type significantly influenced expansion and attributed it to the interaction of flavor compounds with starch during extrusion. Differences may be due to different feed types used (raw vs. pregelatinized starch) and due to feed mc (24% vs. 9–17%).

Effects of feed mc and flavor type on SME are shown in Fig. 3. Maximum SME requirement was for 9% feed mc and was significantly higher ($P < 0.0001$) than those for the 13 and 17% mc. Lower levels of moisture in the feed possibly resulted in increased melt viscosity, causing an increase in the SME requirements. Flavor type also had a significant ($P < 0.012$) influence on SME requirement. The SME requirement of cinnamaldehyde was significantly lower at 9% feed mc. Pregelatinization possibly made flavor-binding reaction sites available on starch molecules, resulting in the significant influence on SME. On the contrary, Kollengode et al (1996a) observed that flavor type had no significant effect on SME when raw starch was used.

The influences of moisture content of pregelatinized starch and flavor type on amount of volatiles retained are shown in Fig. 4. Feed mc influenced significantly ($P < 0.0001$) the amount of flavor retained. Maximum flavor retention was obtained at lower feed mc. In agreement, Kollengode et al (1996a) recommended lower feed mc and higher initial flavor levels for maximum volatile retention. Flavor type also had a significant influence ($P < 0.0001$). The amounts of the flavors retained were of the order: eugenol > 3-octanone > cinnamaldehyde > nonanoic acid. Functional groups of flavor compounds thus seemed to have a significant influence on flavor retention. The alcohol functional group had maximum retention, while the carboxylic group had the least retention. The acidic carboxylic group possibly hydrolyzed the starch backbone resulting in lower retention.

Direct Addition vs. Flavor Injection

Kollengode et al (1996a) reported that injection of flavors into the extruder barrel during extrusion of corn starch resulted in significant increases in flavor retention over preblending of flavor. To

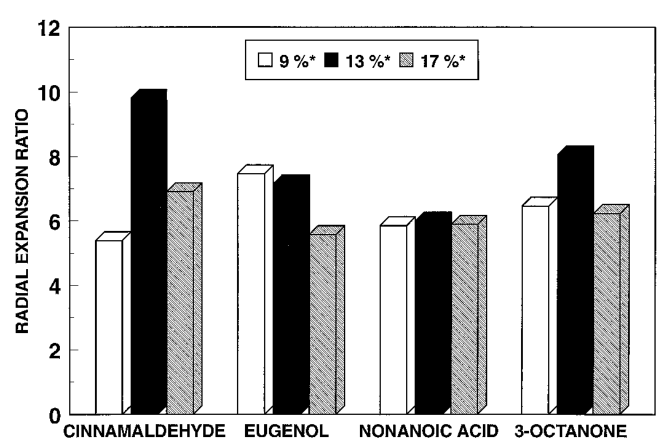

Fig. 2. Effects of initial moisture of pregelatinized starch and flavor compounds on radial expansion ratio of extrudates. * = Average of three replicates.

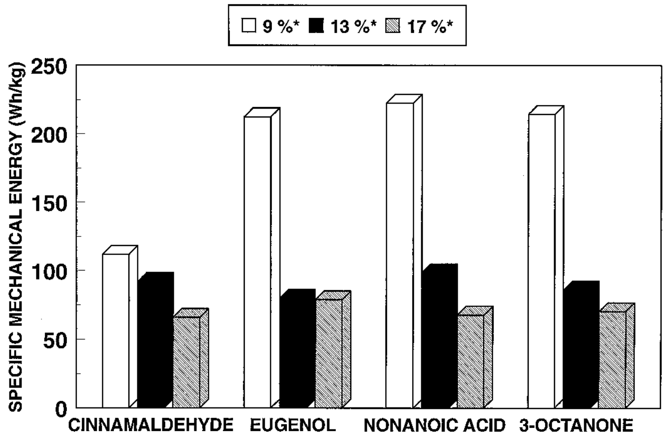

Fig. 3. Effects of initial moisture of pregelatinized starch and flavor compounds on specific mechanical energy (SME) of extrudates. * = Average of three replicates.

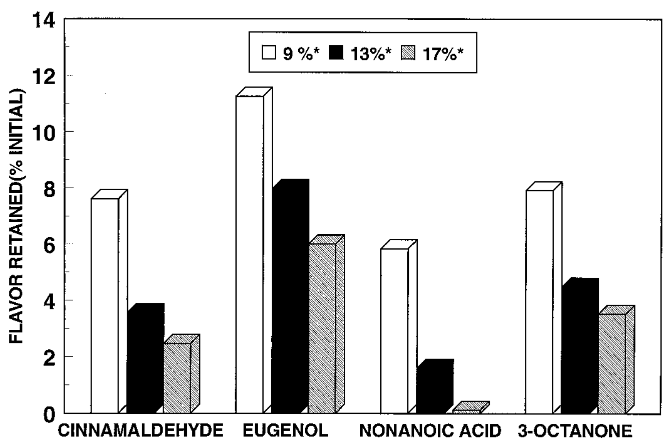

Fig. 4. Effects of initial moisture of pregelatinized starch and flavor compounds on volatiles retention in extrudates. * = Average of three replicates.

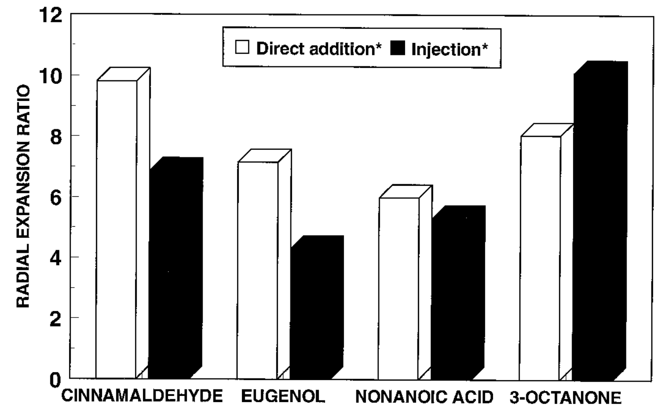

Fig. 5. Effects of flavor addition method on radial expansion ratio of pregelatinized starch (13% mc [db] and 5% initial flavor level [w/w]) extrudates. * = Average of three replicates.

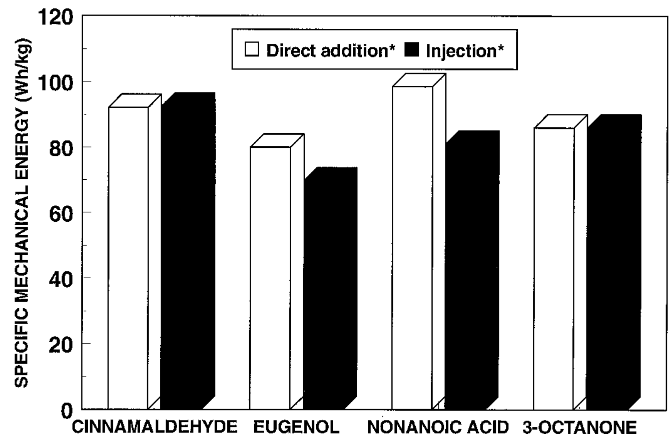

Fig. 6. Effects of flavor addition method on specific mechanical energy (SME) of pregelatinized starch (13% moisture [db] and 5% initial flavor level [w/w]) extrudates. * = Average of three replicates.

verify these findings for pregelatinized starch, 13% feed mc and 5% (w/w) of flavors were used. Preliminary studies (*data not shown*) suggested that flavor compounds could not be injected using 9% feed mc due to excessive torque requirements. At 17% feed mc, the injected flavors back-extruded into the feed hopper. Therefore, 13% feed mc was selected to determine and compare flavor retention by two methods of addition: preblending and direct injection.

Influences of method of flavor addition and flavor type on radial expansion ratio are shown in Fig. 5. Method of addition had no significant effect ($P < 0.0525$) on expansion. Kollengode et al (1996a) reported significant differences due to method of flavor addition during raw starch extrusion. Flavor source had a significant influence ($P < 0.0003$) on expansion of pregelatinized starch. The differences were attributed to the differences in the functional groups of the flavor compounds, which interacted differently with the starch (Kollengode et al 1996a). Blocking over replicates was effective ($P < 0.9711$). Differences between 3-octanone and cinnamaldehyde, or between eugenol and nonanoic acid, were not significant, while all other combinations were significantly different.

The insignificant effect ($P < 0.5829$) of flavor addition method on SME requirements (Fig. 6) was in agreement with findings using raw starch (Kollengode et al 1996a). Flavor compound had a significant effect ($P < 0.0014$). Nonanoic acid, when blended directly with the feed, had the maximum SME while injection of eugenol had the least SME requirement.

The effects of method of flavor addition and flavor compound on volatile retention are shown in Fig. 7. Both had significant influences ($P < 0.0001$ and 0.0006, respectively) on volatile retention. Injection of eugenol or cinnamaldehyde resulted in considerable increases in flavor retention. Retention of nonanoic acid was lower than 3-octanone, irrespective of the method of addition. Kollengode et al (1996a) suggested that increased flavor retention upon injection was due to less severe effects of temperature, shear, pressure, residence time, and possible protection by encapsulation by the molten mass of starch in the extruder as the flavors were injected.

Comparison of Different Flavor Addition Methods

Kollengode et al (1996a) studied the effect of feed mc (15–21%) and initial flavor concentration (1–5%) on flavor retention using response surface methodology. The three methods of flavor addition by internal application techniques (preblending of flavor in raw starch, injection of flavor, and preblending of flavor in pregelatinized starch) were compared to determine whether pregelatinization was a better method to enhance flavor retention.

Flavor type had a significant ($P < 0.0110$) effect on flavor retention (Fig. 8). In general, when feed mc was 17% (db), the retention order was: eugenol > 3-octanone > cinnamaldehyde >nonanoic acid. Flavor retention as influenced by method of addition, is shown in Fig. 8. Method of addition had a significant effect ($P < 0.0001$) on flavor retention. Maximum flavor retention was obtained for all flavors studied when the flavor was injected into the extruder barrel using raw starch. The least flavor retention (5–9%) was obtained when flavor was preblended with pregelati-

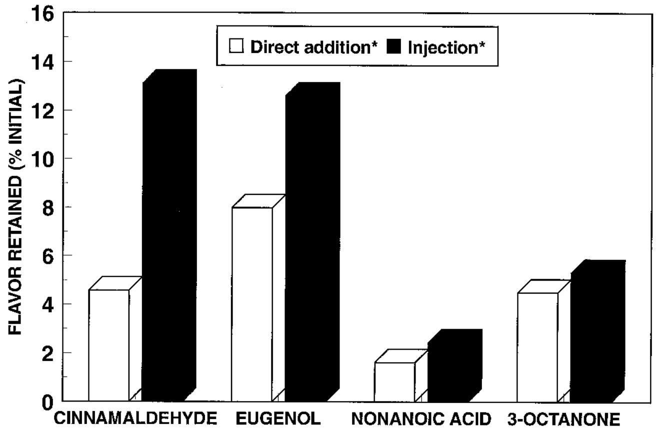

Fig. 7. Effects of flavor addition method on volatiles retention in pregelatinized starch (13% moisture [db] and 5% initial flavor level [w/w]) extrudates. * = Average of three replicates.

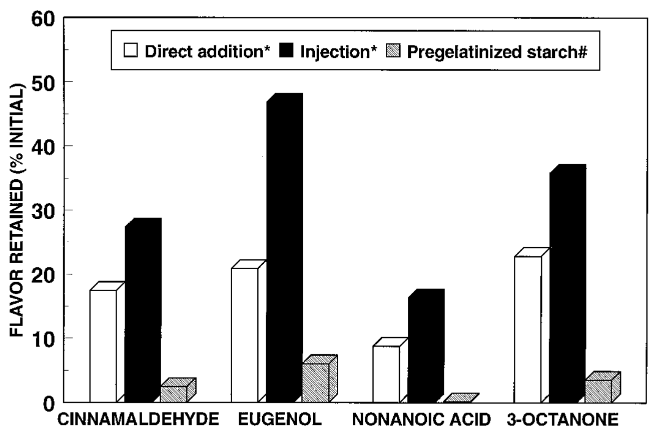

Fig. 8. Comparison of three methods of flavor addition (preblending in raw starch, injection into the raw starch, and preblending in pregelatinized starch) on volatiles retention in extrudates. * = Predicted value. # = Average of three replicates.

nized starch and subsequently extruded. Therefore, use of pregelatinized starch to enhance flavor retention is not recommended. The flavor-loss mechanism in pregelatinized starch is thought to be similar to that of the losses occurring during extrusion of raw starch. Thermal decomposition, oxidation, and polymerization reactions were cited as the reasons rather than volatility of the flavor compounds for flavor losses during extrusion (Kollengode et al 1996a). Pregelatinized starches probably do not provide the appropriate conditions and reactive sites during subsequent extrusion for encapsulation of flavor compounds to a large extent. This probably resulted in flavors prone to flashing-off along with the steam as they exited the die.

CONCLUSIONS

Pregelatinized starch was extruded at 9, 13, and 17% mc to enhance flavor retention by either preblending or by injecting the flavor into the extruder. Moisture content significantly influenced the expansion ratios and SME. Less SME was needed at higher moisture contents. Flavor retention decreased with increased feed mc levels. Injection of flavor into the 13% feed mc starch increased flavor retention when compared to the preblending process. Upon extrusion, flavor retention was lower when added to pregelatinized starch when compared to flavor addition to raw starch.

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[Received October 2, 1996. Accepted February 25, 1997.]