MIGRATION STUDIES OF PACKAGED GRAINS

Plastics are now being used on a large scale for packaging of foodstuffs, pharmaceuticals and water. When food comes in contact with plastics, the high molecular mass polymer does not pose a toxic hazard since it is inert and insoluble in food. However, there is a likelihood for polymer additives, adventitious impurities, such as monomers, catalyst remnants, and residual polymerization solvents and of low molecular mass polymer fractions from plastics to migrate into the food material packed, which may pose hazard over a period of time.

The choice of simulating solvents and test conditions (time-temperature) depends on the type of foods and conditions of use of food products. Food products have been now classified into seven major groups as shown in Table 20. This table has been prepared on the lines of accepted classification of foodstuffs for such purpose. The table also gives suitable simulants to be used for different types of foods.

Table 21 lists the simulants and test conditions (time-temperature) for extractability studies to be carried out as shown above depending on the type of food and conditions of use.

Selection of Samples

Minimum triplicate samples representing the lot/batch have to be selected. Samples in each replicate shall consist of a number of containers (performed or converted products) with nearest exposed area of 1000cm^2. In the case of sealable films representative sample shall be of sufficient size to convert into 2 pouches of size 125 mm width and 200 mm length (inner dimension excluding seal area) and non heat sealable films of size (50cm X 10 cm) to be exposed over both the sides with 1000 cm^2 surface area coming in contact. In case of lids/wads ten pieces are to be sealed to glass bottles of smallest size, in actual use to be placed reverted in position during the test period.

Table 20. Classification of foods and selection of simulants

Sl. No	Type	Description	Examples	Simulants
1	I	Aqueous, non acidic foods (pH>5) without fat	Honey, mineral water, sugar syrups, molasses, skimmed milk, rasgulla, murabba, paste etc	'A' (Water)
2	II	Aqueous, acidic foods (pH<5) without fat	Fruit juices, squashes, fruit chunks or puree or paste, vinegar, jams, jellies, carbonated beverages, lemonade, processed vegetables, preparations of soups, broths sauces, RTS beverages etc.	'B' (3% Acetic acid)
3	III	Alcoholic beverages: i) Alcohol concentration less than 10% ii) Alcohol concentration above 10%	Beer and some pharmaceutical syrups Wine, brandy, whiskey, arrack and other alcoholic drinks	'C[1]' (10% Ethanol) 'C[2]' (50% Ethanol)
4	IV	Oils, fats and processed dry foods with surface fat or volatile oils	Vegetable oils, ghee, vanaspati, cocoa butter, lard, dry products with a surface fat such as biscuits, spice powder, snacks and savory, chocolate, caramels, malted foods egg powder, tea coffee powder confectionery, fried and roasted nuts etc	'D' (n-Heptane)
5	V	Nonacidic foods (pH>5) or high fat and having high moisture content	Butter, bread, pastry, cakes, milk based sweets, ice-cream, moist and fatty confectionery products	'A and D'
6	VI	Acidic foods (pH<5) or high fat and having high moisture content	Pickles, ketchup, cheese, curd, fresh and processed meat and fish products, sauces having fat, frozen foods etc.	'B and D'
7	VII	Dry processed foods without fat	Cereals and pulses, dehydrated vegetable and fruits, dried yeast, corn flakes, salt, sugar, milled products, barley powder, oats, vermicelli, grain etc.,	Only A

Table 21. Simulating solvents for different types of foods and temperature-time conditions

Sl. No	Conditions of use	Type of Food	Migration Test With Food Simulants-Time Conditions (^{0}C/h)				
			Water (A)	3% acet acid (B)	10% alcohol (C^1)	50% Alcohol (C^2)	n-Heptane* (D)
1	High temperature heat Sterilized (Retorting)	I, II, IV, V and V	(121/2)		-	-	(66/2)
2	Hot filled or pasteurized Above 66^{0}C, below 100^{0}C	I, II, IV, V and V	(100/2)		-	-	(49/0.5h)
3	Hot filled or pasteurized below 66^{0}C	I to VI	(70/2)				(38/0.5h)
4	Room temperature filled and stored and also in refrigerated and frozen condition (no thermal treatment in container)	Do	40^{0}C/ 10days				(38/0.5h)
*Heptane extractivity results must be divided by a factor of five in arriving at the extractivity of a food product.							

Preparation of Test Specimen

The containers/pouches/film/lids used were carefully rinsed with water (25-30^{0}C) to remove extraneous materials prior to actual migration test.

In this study simulants such as Distilled water, 3 % Acetic acid, Ethyl alcohol and n-Heptane were used for determining overall migration of constituents from the supplied plastic material by selecting appropriate time-temperature set conditions.

Simulant Quantity

Equal to nominal filling capacity or at least 1 ml/cm^2 of contact area.

Procedure

The number of samples constituting 1000 sq.cm per replicate was exposed to preconditioned solvent (1000 ml) in a glass beaker, covered with glass plates and kept at the test temperature for the stipulated period in an oven. In case of PP cups and HDPE jars the number of samples constituting nearly 1000 sq.cm per replicate were filled with the preconditioned solvent. At the end of the test period, the sample was removed and the solvent in each replicate was concentrated to about 50 ml by evaporation/distillation. This concentrate along with three washings was transferred to a tared stainless steel dish, and evaporated to dryness at 100±5°C in an oven. After drying, the dishes were cooled for 30 minutes in a desiccator, and weighed to nearest 0.1 mg. Blank shall also be carried out without the sample for adjustment, if necessary.

$$\text{Amount of extractive (Ex)} = \frac{M}{A} \times 100 \quad \text{mg/dm}^2, \text{ and}$$

$$\frac{M}{V} \times 100 \quad \text{ppm}$$

where

M = mass of residue in mg minus blank value

A = total surface area in cm^2 exposed in each replicate, and

V = total volume in ml of simulant used in each replicate

II. PROGRAMME OF WORK

Global Migration tests on different types of plastic packaging materials namely HDPE woven sack, PP woven sack, LDPE film, PP cup and HDPE jars were carried out as per BIS method with different food simulating solvents as per the required end use.

Reagents

Distilled water, 50% ethanol, 3% acetic acid and n-heptane

Equipments

Analytical balance, beakers, Hot air oven, Thermometer, Stainless steel dishes, Retort vessel, Hot plate.

RESULTS AND DISCUSSION

Different types of plastic packaging materials were evaluated by global migration test with food simulants namely Distilled water, 3 % Acetic acid, 50 % Ethanol and n-heptane at different test conditions of time and temperature depending upon the end use. As shown in the Table 22 the average of triplicate values for global migration ranged 0.78 mg/dm^2 and 0.42 ppm in HDPE jars to 2.44 mg/dm^2 and 5.40 ppm in HDPE woven sack. The average values for global migration with different simulants are well within the limits of maximum 10.00 mg/dm^2 and 60 ppm specified in BIS specifications.

CONCLUSIONS

The global migration values for the samples tested with different food simulants under the specified test conditions of time and temperature are well below the specified maximum limits as per BIS specifications as shown in table

REFERENCES

i. IS: 9845 - 1998: "Determination of Overall migration of constituents of plastic materials and articles intended to come in contact with foodstuffs" – Method of analysis.

ii. IS: 10141-1982: "Positive list of constituents of polyethylene in contact with foodstuffs, pharmaceuticals and drinking water."

iii. IS: 10146- 1982: "Specification for polyethylene for its safe use in contact with foodstuffs, pharmaceuticals and drinking water."

iv. IS:10171 - 1982: "Guide on suitability of plastics for food packaging"

v. IS: 10909 - 1984 "Positive list of constituents of polypropylene and its copolymers for its safe use in contact with foodstuffs, pharmaceuticals and drinking water (First Revision)

vi. IS: 10910 - 1984 "Specification for Polypropylene and its copolymers for its safe use in contact with foodstuffs, pharmaceuticals and drinking water."

Table 22. Overall migration values for different plastic packaging materials

Sl. No.	Packaging materials	Intended use	Global Migration Test with Food Simulant (Temp/Time)	Amount of extractives	
				Mg/dm^2	Ppm
1	HDPE Woven sack	Grain storing	Dist.water (40^{0}C/10 d)	2.44	5.4**
2	PP Woven sack	Grain storing	Dist.water (40^{0}C/10 d)	1.39	3.2**
3	HDPE Jars	Oil/ghee packing	n-Heptane (38^{0}C/0.5 h)	0.078	0.42
4	PP Cups	Hot coffee/ tea contact	Dist.water (100^{0}C/2 h) n-Heptane (66^{0}C/0.5 h)	0.38 1.33	3.1 16.6
5	LDPE Film	General food contact	Dist..water (40^{0}C/10 d) 3 % Acetic acid (40^{0}C/10 d) 10% Ethanol (40^{0}C/10 d) 50% Ethanol (40^{0}C/10d) n-Heptane (38^{0}C/0.5 h)	0.20 0.32 0.22 0.27 0.17	2.0 3.2 2.2 2.7 1.7

*Average of triplicate
** ppm based on 50 kg capacity bag
Limit: 10 mg/dm^2 and 60 ppm
Opinion: The The materials tested conform to BIS specifications .

TESTING OF WOVEN SACK MATERIALS FOR ICPE

PROGRAMME OF WORK

Water vapour transmission rate (WVTR)

WVTR of High density polyethylene(HDPE) and polypropylene(PP) woven sack material was tested a per BIS method. 50 sq.cm of the packaging material in circular shape was sealed to an aluminium dish containing anhydrous calcium chloride as desiccant and exposed to 92 % RH/38^{0}C. The permeated water vapour was measured gravimetrically and the results are expressed in g/sq.cm/day under 90 % RH gradient at 38^{0}C.

Air Resistance (porosity)

Air Resistance (porosity) was measured as per British standards or ASTM method. The apparatus consists of a cylinder half filled with lubricating oil. A graduated cylindrical pipe which exactly fits to the cylinder slides in the cylinder. On the top of the pipe the sample is secured with the help of screws and gaskets (to avoid leakages) exposing an area of 1 sq.in. The pipe is taken up and let down slowly. The pipe slides down due to gravity compressing the head space air. Depending on the resistance offered by the packaging material air gets depleted and pipe slides down. The result is expressed as time taken to deplete 100 cc of air by 1 sq.in of the material.

Coefficient of friction

Coefficient of friction was done for three packaging materials namely HDPE, PP and jute woven sacks. For this, the test material was clipped to a wooden board. Another sample of the same material is wrapped on a rectangular metal piece (used as a standard weight)) and kept on the board. The board is slowly raised at one end in anticlockwise direction to note the angle (θ) at which the weight slides on the board. The results are expressed as tan θ.

Moisture sorption data

The moisture sorption behaviour of wheat and rice used for the storage studies was determined by exposing known quantity of the product to different RH's ranging between 11 and 92 % built in desiccators using appropriate saturated salt solutions at 27 $\pm$ 1^0C(Lopez.A et.al). The samples were weighed periodically till they attained constant weight or showed the signs of mould growth. The equilibrium moisture content was calculated by adding / subtracting the percentage gain/loss to the initial moisture content of the product.

RESULTS AND DISCUSSION

WVTR: As can be seen from table 23, the HDPE and PP woven sack materials have high WVTR in the range of 2167-2645 g/sq.m./day under 90 % RH gradient at 38^0C

Air Resistance (porosity): Since there were openings in all the three materials tested , time required to displace 100 cc of air by 1 sq.cm of the material could not be determined as the displacement was instantaneous

Coefficient of friction: The coefficient of friction was maximum for jute followed by PP and HDPE

Sorption data: As the critical RH is 65 % for both rice and wheat to store these products at and below 65 % RH a moisture barrier is not required to protect the product from moisture ingress. So unlined HDPE, PP or jute sacks are sufficient. But to store them above 65 % RH a moisture proof polyolefin liner for the woven material will be essential. (Table 24)

Conclusion: WVTR of unlined PP and HDPE woven sacks are high as that of jute. Breathability of PP and HDPE woven sacks are comparable to that of jute sacks. Higher value of coefficient of friction in PP woven sacks will help in stacking.

References

1. Bureau of Indian standards (1960). Method of sampling and test for paper and allied products is: 1060 (part ii) new Delhi.
2. British Standards BS 2925
3. American Standards for Testing Materials ASTM D726
4. Lopez.A, Pique. M.T, Tasius. J. (1995). The hygroscopic behaviour of hazelnut J.Food. Engg.

Table 23: Properties of packaging materials.

Sl. No.	Packaging material	WVTR Outer	WVTR Inner	Air porosity	Coefficient of Friction θ (tan θ) AL. AL	AL AC	AC AC
1.	HDPE	2167	2557	Instan-taneous	22^0 (0.4040)	20^0 (0.3640)	17^0 (0.3057)
2.	PP	2658	2645	-do-	28^0 (0.5317)	21^0 (0.3839)	20^0 (0.3640)
3.	Jute	-	-	-do-	$>44^0$ (>0.9657)	$>44^0$ (>0.9657)	$>44^0$ (>0.9657)

AL: Along the bag

AC: Across the bag

Table 24: Moisture sorption data on wheat and rice

Sl. No.	% RH	EMC (dry wt.)		Remarks
		Wheat	Rice	
1.	11	7.35	5.91	Very hard
2.	22	8.90	8.27	Very hard
3.	33	10.40	9.98	Good
4.	44	11.92	11.76	Good
5.	56	13.46	13.10	Good
6.	64	14.14	13.88	Good
7.	75	17.39	16.38	Musty odour
8.	86	20.19	19.20	Mould
9.	92	23.77	21.46	Mould
	IMC (as is)	10.35	11.12	
	ERH	40%	50%	
	CMC (as is)	13%	12.5%	
	CRH	65%	65%	