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Preliminary Experiments on Development of a Technology for Citrus Waste Utilization. *I*. Potential Nutrients of Pakistani Sweat Orange Products

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Different sweat orange products like juice, peel, coarse peel, fines of peel, lime treated peel, molasses extracted peel, juice sacs, molasses and skin (peel without juice sacs) etc. were developed at laboratory scale and dried in an electric oven. Maximum crude protein (7.5%), crude fat (5.27%) and ash (5.53%) contents were recorded in dried juice sacs. Moisture content of different dried products ranged between 7.44-13.36%. Untreated peel contained highest amount of ascorbic acid (408.75 mg100g^{-1}) and beta-carotene (25.98 mg g^{-1}). All the products contained more than 1% pectin and its fractions showed some variation.

Key words: Waste utilization, Sweat orange, Nutritive value

Introduction

Citrus is one of the largest fruit crops grown in Pakistan and the total area under production and the produce are 194.4 thousand hectares and 2002.6 thousand tons, respectively (Anon 1997). A large part of produce is used as table fruit and recently a few national and international food industries have started producing pure citrus juice, ready to drink juices and marmalades etc. Strong national and international future demand for citrus products will provide stimulus to maintain increasing levels of production. When citrus is processed for juice, the remaining residue (peel, membranes, essential oils and seed etc.) amounts to about 50% of the whole mass. The residue left after juicing is the source of numerous by-products like dried citrus peel, molasses, flavonoids, limonene, pectin and seed oil (Cadwallader *et al* 1989; Huet 1991; Braddock and Cadwallader 1992; Braddock 1995; Fox 1995).

Recovery of pectin from dried peel mass has a potential in food products like jams, jelly and marmalades. Limonene is a valuable by-product of citrus and can be converted into terpineol, which is more valuable than limonene (Waters 1995). Citrus is an especially rich source of two important flavonone glycosides i.e. hesperidin and naringin. Pounden and Frank (1965) demonstrated that 15 pounds of dried citrus pulp or 50 g of hesperidin complex fed daily to dairy cows for 5 months reduced the incidence of mastitis caused by *Streptococcus galactiae*. This paper presents results on the potential nutrients of different peel products of Pakistani sweat organge.

Materials and Methods

Sweat oranges (*Citrus sinensis* L. var. Mosambi) about 100 kg was purchased from local market. The samples were washed throughly under running tap water, sorted and the juice was manually extracted with juice extractor (Fig 1). The juice was converted into squash. The peel and membranes etc. were cut with the help of vegetable cutter (Model VA20, Nihon Choriki Seizoco Ltd. Japan). The cut peel was divided into two lots (Fig 1). One lot was treated with 0.5% lime at room temeprature for 3 h and pressed in a hydraulic press (Kiya Seisaku Shu Ltd., Tokyo, Japan) at room temperature to extract liquor. This liquor, concentrated to 65°Brix by heating, , is called citrus molasses. The untreated peel was further divided into two lots. Juice sacs were removed with stainless knives in one lot. The second lot, from which juice sacs and molasses were extracted, was dried in an electric oven (Binder, Germany) at 70 ± 2°C. The dried peel with sacs was sifted through 14 mesh and was divided into fines and course peel.

Moisture, protein, fat, beta-carotene, ascorbic acid and ash contents were determined using standard AOAC mehods (1984) as under. Moisture content was determined using air oven method (105 ± 1°C). The crude fat was estimated using Kjeldahl method (N x 6.25). Muffle furnace (550°C) was used for ash contents. Titrimetric method was applied for ascorbic acid estimation using *2,6* dischlorophenol indophenol dye to

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a light pink end point. Acetone extract was used to determine beta-carotene by taking optical density at 410 nm in a Shimadzu spectrophotometer (Model UV-120-02). Standard curve was also drawn using beta-carotene solution in acetone (Fig 2).

Total pectin and different fractions (water, ammonium oxalate and alkali soluble) were determined by developing colour complex with sulphuric acid and carbazole reagent (Rouse and Atkins 1955). The intensity of the red colour was read in a spectrophotometer at 525 nm. A standard curve was drawn using 125.5 mg of monogalaturonic acid monohydrate previously dried over phosphorus pentoxide at room temperature (Fig 3). Working standards covering a range of 10-70 μg of galacturonic acid were prepared. The means and CV were determined (Steal and Torrie 1980).

Results and Discussion

Table 1 shows the proximate composition of different peel products. The moisture content ranged between 7.4% (dried skin) to 90.17% (pure juice). Highest crude fat (5.27%) was recorded in juice sacs followed by lime treated peel and lowest (0.44%) in molasses. Maximum protein contents (7.53%) were recorded in juice sacs followed by dried skin (7.0%) and minimum (1.1%) in juice. The ash content ranged between 0.007% (juice) to 5.53% (dried juice sacs). Peel without molasses contained maximum ascorbic acid (408.75 mg $100g^{-1}$) and maximum beta-carotene (25.98 mg g^{-1}) was found in juice sacs.

On similar studies Ammernan *et al* (1966) have reported protein (6.74%), ether extract (4.1%), crude fiber (13.44%) and nitrogen free extract (NFE-70.6%) while analyzing 1,728 samples from Florida, USA, covering a wide area and found some variations due to locations. These values closely agree to our results prsented in this paper. He also reported that for each 1% increment of citrus seeds substituted for peel plus rag, ether extract was increased by 0.39% and protein 0.09% in the final product.

Looking at the data on ascorbic acid and beta-arotene, it is evident that all the products contained substantial amounts of these vitamins. Juice sacs contained more ascorbic acid and beta-carotene than other products with very few exceptions proving their potential for useful utilization. Incorporation of peel products for the improvement of egg yolk colour has been reported (Hinton 1972). The results reported by Ammerman *et al* (1966) and Kesterson and Braddock (1976) on the composition of citrus pulp are also in agreement with the present results.

Pectin is a polymer with many (a-1, 4-liked D-galacturonic acid moieties. But it also contains neutral sugars such as L-arabinose, D-galactose and L-rhamnose. Pectin is ubiquitous polysaccharides in the albedo (mesocarp) and in the edibles portion (endocarp) of citrus fruits and is primarily a part of the structure. When citrus fruits are processed into juice, some of the pectin is disrupted and becomes soluble in the serum. Large macroscopic fragments containing pectin are considered pulp.

It was found that high methoxyl pectin, pectates, pectinates

Table 1

Nutrient composition (%) of different sweat orange products.

Sample	Proximate (%)				Vitamins	
	Moisture	Fat	Ash	Protein	Ascorbic acid (mg $100g^{-1}$)	β-carotene (mg g^{-1})
Whole fruit (F)	85.34	*	5.03	*	29.8	5.31
Juice (F)	93.87	*	.01	1.09	22.5	7.98
Citrus peel (F)	80.84	3.63	4.94	5.95	41.3	13.28
Citrus peel**	13.36	3.44	4.61	4.55	266.3	25.0
Coarse fines of coarse peel	11.11	1.85	6.18	7.35	172.5	6.81
Lime treated peel (0.5%)(F)	81.23	3.78	5.17	5.16	26.3	6.64
Molasses extracted peel	7.64	1.67	4.54	6.3	408.75	25.98
Molasses	40.77	0.44	4.70	3.75	*	17.18
Skin	7.44	3.22	5.22	7.0	52.5	-
Juice Sacs (F)	85.93	5.27	5.53	7.53	60.0	-

* Samples were not analyzed;** Peel represents skin, juice sacs, seeds and pulp remaining after juice extraction; F, Fresh without drying.

Table 2
Distribution of different pectin fractions in sweat orange products

Sample	Pectin-% (Anhydro-galacturonic Acid			Total Pectin (%)	% of total pectin		
	1	2	3	1+2+3	1	2	3
Whole fruit	0.33	0.41	0.41	1.15	28.7	35.65	35.65
Juice	0.24	0.15	0.12	0.51	47.1	29.4	23.5
Citrus peel*	0.29	0.41	0.41	1.11	26.1	36.9	36.9
Citrus peel coarse	0.37	0.42	0.41	1.20	30.8	35.0	34.2
Fines of coarse peel	0.32	0.41	0.41	1.14	28.0	36.0	36.0
Lime treated peel (0.5%)	0.28	0.41	0.41	1.10	25.4	37.3	37.3
Molasses extracted peel	0.41	0.42	0.41	1.24	33.06	33.9	33.96
Molasses	0.25	0.41	0.41	1.07	23.4	38.3	38.3
Skin	0.38	0.42	0.41	1.21	31.4	34.7	33.9
Juice sacs	0.25	0.41	0.41	1.07	23.4	38.3	38.3

*, Peel represents skin, juice sacs, seeds and pulp remaining after juice extraction; 1, High methoxyl pectin; 2, Pectates and pectinates; 3, Protopectin

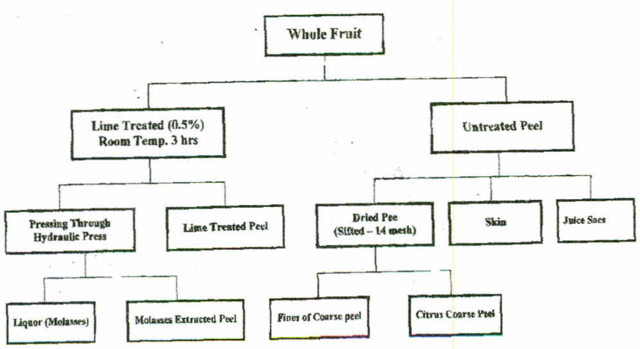

Fig 1. Preparation of different orange products

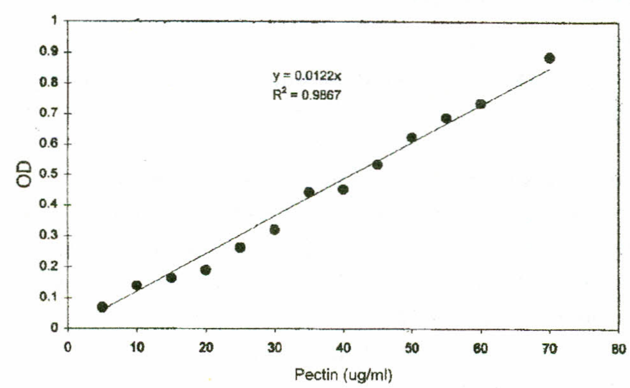

Fig 3. Standard curve for pectin

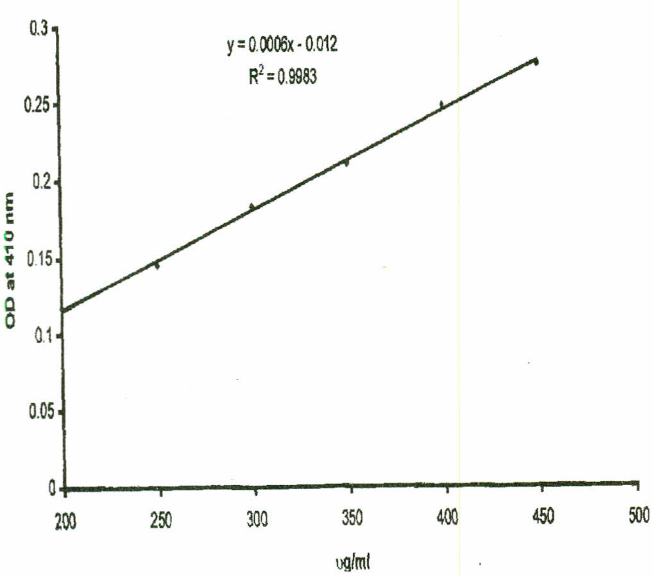

Fig 2. Standard curve for β-carotene

and protopectin varied from 0.24 - 0.41, 0.15 - 0.42, 0.12 - 0.41% respectively (Table 2). Maximum total pectin (1.24%) was found in citrus peel after liquor extraction followed by citrus peel (1.20%) without extraction of molasses. Klanvons *et al* (1994) have reported 4.5% and 4.1% pectin in commercial orange juice cloud and also reported that protopectin (about 15%) was insoluble in urea-citrate buffer (pH 2.5). Braddock((1995) has reported calculated amounts of pectin as 1.3 and 1.0 kg box^{-1} of oranges and grapefruit, respectively. The pectin has many commercial applications in food and medicines where gelling is needed in the final product because pectin and its fractions are one of the best natural gelling ingredients. They also act as anticonstipatives and can absorb injurious materials from stomach/intestine.

Variations in nutrient contents of citrus pulp products have been attributed to differences in dehydration procedures, source of fruit, type of operations, and seed content (Ammerman and Henry 1968; Hendrickson and Kesterson 1976) and varieties (Pascual and Carmand 1980a). Lanza

(1984) has also emphasized the importance of variety of fruit in influencing the chemical composition of citrus pulp.

The substantial amount of different nutrients in the samples and expected increase in future citrus processing in Pakistan provides sufficient justification for the development of technology for citrus by-products and their utilization as feed and food.

References

Ammerman C B, Easley J F, Arrington L R, Martin F G 1966 Factors affecting the physical and nutrient composition of dried citrus peel. *Proc Florida State Hortic Soc* **79** 223.

Ammerman C B, Henry P R 1968 *Citrus and Vegetable Products for Ruminants* . Personal communication. Deptt Ani Sci, Uni Florida, Gainesville, Florida, 32611.

Anon 1997. *Agricultural statistics of Pakistan.* Ministry of Food, Agriculture and Cooperatives (Government of Pakistan), Islamabad pp. 54.

AOAC 1984 *Official Methods of Analysis.* Association of Official Analytical Chemists, 14th ed, Washington, DC.

Braddock R J 1995 By-products of citrus fruit. *Food Technol* **49** 74 & 76-77.

Braddock R J, Cadwallader K R 1992. Citrus by-products manufacture for food uses. *Food Technol* **46** 103-110.

Cadwallader K R, Braddock R J, Parish M E, Higgis D P 1989. Bioconversion of (+) limonene. *J Food Sci* **54** 1241.

Fox K L 1995 *Preliminary Pulp Recovery.* Personal communication. FMC Corp, Lake Alfred, Florida.

Hendrickson R, Kesterson J W 1976 Citrus pulp with and without seeds. *Proc Florida State Hortic Soc* **79** 248.

Hinton C F 1972 Reflectance colorimetric evaluation of carotenoid pigments in broilers and egg yolks. PhD Thesis, Univ of Florida Florida USA.

Huet R 1991 Les huiles essentielles d argumes. *Fruits* **46** 501-513, 551-576.

Kesterson J W, Braddock R J 1976 *By-Products and Speciality Products of Florida Citrus.* Bull No 784 Agric Experiment Stations, Lake Alfred, IFAS, Univ Florida.

Klanvons J A, Bennett D, Raymond, Sadie H V 1994 Physical/chemical nature of pectin associated with commercial orange juice cloud. *J Food Sci* **59** 399.

Lanza A 1984 Dried citrus pulp in animal feed. *Proc Int Symp,* ed Hollo J, Sept 9-11, 1982, Budapest, Hungary, Elsevier, Amsterdam.

Pascual J Martinenz, Carmond J Fernandez 1980a. Composition of citrus pulp. *Animal Feed Sci Technol* **5** 1.

Pounden W D, Frank N A 1965 Influence of citrus pulp products on *Streptococcus galactiae* mastitis in cattle. *J Amer Vet Med Assn* **146** (11) 1304.

Rouse A H, Atkins C D 1955 *Pectin Esterase and Pectin in Commercial Citrus Juices by Methods Used at the Citrus Experiment Station.* Tech Bull No. 570. Agric Exp Stn, Univ Florida, USA.

Steel R G D, Torrie J H 1980 *Principles and Procedures of Statistics.* McGraw Hill Publ Co Inc New York.

Waters R 1995 *Pulp Recovery Systems.* Private communication. Automatic Machinery & Electronic Co. Winter Haven, Fla.