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Citrus wastes: Composition, functional properties and utilization

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ABSTRACT

Processing of citrus juice generates large amounts of wastes such as peels, segment membranes and seeds. Utilization of wastes is necessary because of economical and environmental reasons. The present work aimed at extracting the pectic polysaccharides of citrus peels so that the gelling properties are utilized, while the peel fibers are present. After juice extraction the citrus peels were macerated, leached in hot water, pressed and the resultant mash was mixed with acidified alcohol. The mixture was incubated for 22 hrs, filtered, washed with ethanol, dried and finally milled. This product was designated as fiber-pectin. Chemical and physical properties of fiber-pectin were studied.

The results showed that the yield of lime fiber-pectin and orange fiber-pectin was 9.7 and 8.6%, respectively, based on wet peel weight. A higher amount of anhydrogalacturonic acid was found in lime fiber-pectin (38.21%) than in orange fiber-pectin (29.62%). Degree of methylation of both types of fiber-pectin was 70.38 and 65.36%, respectively. In lime fiber-pectin 40% of the total pectin was water-soluble, while in orange fiber-pectin this fraction represented only 16%. Lime fiber-pectin was characterized by higher swelling (37.66 ml H₂O/g fiber) and hydration capacity (17.77 ml H₂O/g fiber) than orange fiber-pectin, being 21.23 and 13.40 ml H₂O/g, respectively.

For both types of fiber-pectin the maximum breaking gel strength was found at 60% sucrose concentration, compared to 65% sucrose concentration for com-

mercial citrus pectin. Breaking gel strength of orange fiber-pectin was 27.9% of that displayed by lime fiber-pectin. Color, taste and texture of strawberry jam made by lime fiber-pectin were comparable to jam produced with commercial pectin.

In conclusion, processing of fiber-pectin is a promising process. It is simple, inexpensive and yields a product having gel properties comparable to commercial pectin.

INTRODUCTION

Because of economical and environmental reasons there is a growing interest in the production of by-products from citrus wastes resulting from juice processing. Citrus peels have been considered the main source for pectin production. Among citrus peels, lime peels are the most common and valuable source of commercial pectin due to the high yield and quality of pectin. Pectin yield from dry lime peel ranges between 20 and 35% (ENDREB, 1993), which is higher than that obtained from apple pomace (15-20%). The highest gel strength and viscosity of pectin is obtained from lime peels followed by apple pomace and lemon peels (ROUSE and CRANDALL, 1978; MAY, 1990).

The industrial extraction of pectin is based on converting the protopectin into high molecular-weight soluble pectin by splitting the acid-labile linkages between pectin molecules and hemicellulose (FRY, 1986). This is normally achieved through acid-hydrolysis using mineral acids in aqueous medium at pH 1.5 - 3.0 and temperature 60 - 100°C (VORAGEN and PILNIK, 1994).

The crude pectin extract is concentrated and precipitated with alcohol. The precipitate is separated and washed several times to remove the alcohol-soluble components such as acids, sugars, pigments, polyphenols, and heavy metals. It is then vacuum-dried and milled, yielding an unstandardised pure pectin. The starting material used for industrial pectin extraction are most frequently dried citrus peels or dried apple pomace. Previous heat treatment is necessary to inactivate the native pectin degrading enzymes (MAY, 1990). ROUSE and CRANDALL (1978) found that the jelly grade of pectin extracted from leached fresh lime peel was higher than that extracted from dried peel. SILIHA (1993) extracted freshly leached lime peels with acidified alcohol at 42°C for 24 hr. The pectin solubilized from the cell walls is instantly precipitated in the alcoholic medium, then the mixture is filtered and washed several times with alcohol and finally dried. Due to the acid extraction of the pectin and to the presence of other peel fibers, this product has been designated as fiber-pectin (SILIHA, 1993).

Our objectives were to investigate the chemical and physical properties of fiber-pectin extracted from lime and orange peels and to evaluate its potential utilization for jam production.

MATERIALS AND METHODS

Preparation of lime and orange peel

Lime (*Citrus aurantifolia*) and orange (*Citrus sinensis*) fruits were purchased from the local market in El-Salhya, El-Sharkia, Egypt. The flavedo layer

was abraded using a carborandum. After juice extraction the peels were ground in a meat mincer to particle size 2-3mm, leached in hot water (ratio of peel to water was 1:3) at 90°C for 10 min to remove the soluble solids and to inactivate the native enzymes. The mash was cooled by dipping in cold water and pressed to remove excess water.

Preparation of fiber-pectin

Lime and orange peel mash was mixed with hot acidified ethanol 96% (60°C) at a ratio of 1:2. The pH of the mixture was adjusted during extraction at 1.8 by hydrochloric acid. After cooling to 42°C it was incubated at the same temperature for 22 hrs, then filtered through a Büchner funnel and washed twice with 70% ethanol, twice with 96% ethanol and finally with acetone. The resultant fiber-pectin was allowed to dry at room temperature and milled in Retsch mill to particle size 0.5 mm.

Fractionation of fiber-pectin

The polysaccharides of fiber-pectin were sequentially fractionated into water-, oxalate-, acid-, and alkali-soluble pectin according to the method outlined by SILIHA et al. (1996).

Analytical Methods

Moisture, protein and mineral contents were measured according to the AOAC (1984). Anhydrogalacturonic acid content (AGA) was determined according to the method described by BLUMENKRANTZ and ASBOE-HANSEN (1973). Degree of methylation (DM%) and degree of acetylation (DAc%) were analyzed, using high performance liquid chromatography (VORAGEN et al., 1986). Neutral sugar contents were analyzed, using the method of ENGLYST and CUMMINGS (1984). Swelling of fiber-pectin was determined, using the bed volume procedure of KUNIAK and MARCHESSAULT (1972). The water-holding capacity (WHC) was estimated according to BORROTO et al. (1995). Determination of breaking gel

strength of fiber-pectin gels was performed using a Herbstreith-pectinometer according to ZEDLER (1983). Breaking strength was expressed as Herbstreith-pectinometer units (HPU).

Jam production

Deep-frozen strawberries (variety Elsanta) were cut into halves and mixed with sugar 1:1.12. The mixture was heated in an open cooker with continuous stirring until the total soluble solids reached 63°Brix, then fiber-pectin or commercial pectin (0.5%) suspended in 300 ml water was added. The mixture was heated again until the total soluble solids reached 59°Brix, then citric acid (50% w/v) was added to adjust the pH at 3.0. The hot jam was poured in glass jars (350ml), sealed and stored at 20°C.

Sensory evaluation

Color, taste, texture and preference of the jams were evaluated after 24 hrs by 10 panelists using a hedonic scale for color and taste with the following characteristics: excellent 5, good 4, average 3, fair 2, and bad 1. As for the texture, the hedonic scale was as follows: too hard 5, hard 4, suitable 3, soft 2, and too soft 1. The hedonic scale for the preference was: very pleasant 6, pleasant 5, mildly pleasant 4, mildly unpleasant 3, unpleasant 2, and very unpleasant 1. The scores given were statistically analyzed using L.S.D. test (GOMEZ and GOMEZ, 1984).

RESULTS AND DISCUSSION

Chemical properties

The yields of fiber-pectin obtained from wet lime and orange peels were 9.7 and 8.6%, respectively. On a dry weight basis the yield was 68.59 and 73.63%, respectively. Due to the presence of fiber materials other than pectin, the yield of fiber-pectin is higher than that reported in literature for pure pectin extracted from dried peels of lime, lemon and grapefruit and from apple pomace

Component	Lime	Orange
Moisture %	4.90	4.51
Protein %	4.98	5.27
Ash %	2.03	3.73
Na ⁺	20.84	53.00
K ⁺	297.50	432.50
Ca ⁺⁺	515.43	1261.73
Mg ⁺⁺	20.34	37.90

Table 1: Water, protein and mineral contents of lime and orange fiber-pectin. Minerals are expressed as mg/100g dry weight.

Component	Lime	Orange
AGA %	38.21	29.62
DM %	70.38	65.36
DAc %	12.28	14.95
Neutral sugars*	28.97	33.43
Rhamnose	1.17	1.79
Fucose	0.29	0.29
Ribose	0.01	0.10
Arabinose	9.25	5.73
Xylose	2.59	1.44
Mannose	1.74	1.64
Galactose	4.42	5.47
Glucose	9.54	16.97

*as mg/100g fiber-pectin.

Table 2: Sugar composition of lime and orange fiber-pectin.

(MAY, 1990). Table 1 shows some chemical constituents of lime and orange fiber-pectin. Orange fiber-pectin contained higher ash content than lime fiber-pectin. The prevailing mineral was calcium representing 33.8% and 25.4% of the ash obtained from orange and lime fiber-pectin, respectively. In pure apple pectin NEIDHART et al. (1996) found 170mg calcium per 100g pectin, while in two commercial lemon pectins KRAVTCHENKO et al. (1992) reported 360 and 740mg calcium. Similarly, potassium content in lime fiber-pectin was 297.5mg/100g which is comparable with that found in lemon pectin (270mg/100g). Lime fiber-pectin was characterized by higher AGA content (38.21%) than orange fiber-pectin (29.62%), representing both the wa-

ter-soluble pectin and the pectin firmly bound to the hemicellulose and cellulose fractions (Table 2). Fiber-pectin from lime and orange are highly methylated, with maximum value for lime fiber-pectin. This is in agreement with data generally observed for commercial pectins (AXELOS et al., 1989; KRAVTCHENKO et al., 1992; NEIDHART et al., 1996). DAc was found in the range of 12 and 14% for lime and orange fiber-pectin, respectively (Table 2). The DAc greatly differ from those reported in literature for commercial pectins (VORAGEN et al., 1986; KRAVTCHENKO et al., 1992; NEIDHART et al., 1996). Similar findings have been reported in alcohol-insoluble solids prepared from apples (SCHOLS and VORAGEN, 1994) and cherries (SILIHA and GIERSCHNER, 1998). Since fiber-pectin contains all fractions of cell wall polysaccharides, it is assumed that insoluble pectic polysaccharides and/or hemicellulose fraction are highly acetylated. SCHOLS and VORAGEN (1994) stated that hemicelluloses are partly esterified with acetic acid. Neutral sugars composition of lime and orange fiber-pectins are presented in Table 2. Total neutral sugars content accounted for 28.97 and 33.43% in-lime and orange fiber-pectins, respectively. The observed values are higher than that reported for

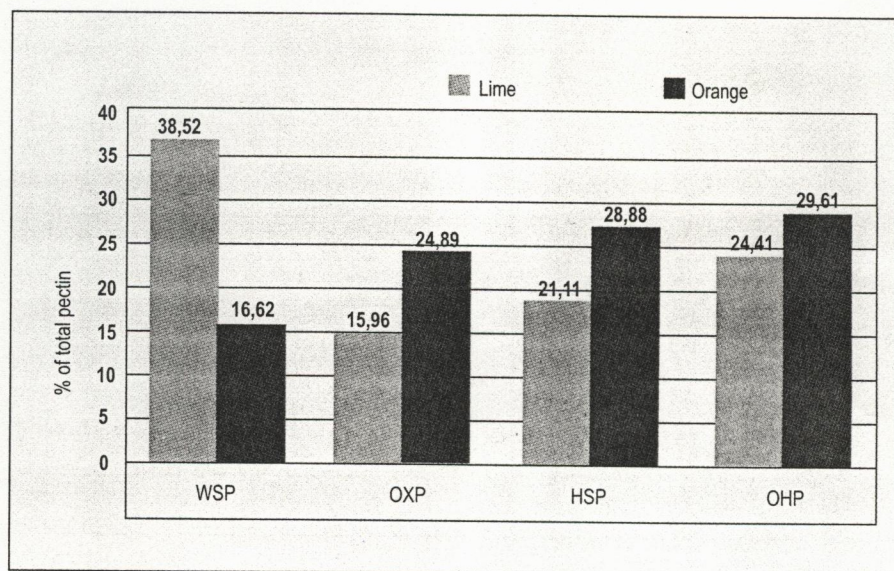


Fig. 1: Distribution of pectin fractions in lime and orange fiber-pectin.

commercial pectins, due to the presence of fibers other than pectin in the fiber-pectins. The predominating sugars were glucose, arabinose and galactose, whereas low contents of rhamnose, xylose and mannose were found. Fucose and ribose were only present in trace amounts. Fiber-pectins of different origin varied in the content of individual neutral sugar. Lime fiber-pectin contained higher amounts of arabinose, xylose and mannose and lower amounts of the other sugars than orange fiber-pectin.

For the understanding of the functional properties of fiber-pectin, it is essential

to know how the pectin fractions are distributed regarding the solubility. Therefore, fiber-pectin was fractionated according to its solubility in water (WSP), ammonium oxalate (OXP), acid (HSP), and alkali (OHP) (Fig.1). The total pectin of both types of fiber-pectin was found to be equal, making up 53.43 and 53.19% of the lime and orange fiber-pectin, respectively. Water-soluble pectin which is of importance with respect to gel formation and viscosity represented the major pectin fraction in lime fiber-pectin (38.52%), while in orange fiber-pectin it made up only 16.62%. In contrast, the OXP fraction of

Component	Lime				Orange			
	WSP	OXP	HSP	OHP	WSP	OXP	HSP	OHP
AGA %	66.17	74.11	40.82	25.81	59.60	75.36	43.38	29.38
DM %	89.60	63.60	76.39	-	77.51	66.63	65.55	-
DAc %	4.83	2.37	7.04	6.36	6.69	2.57	6.74	6.10
Neutral sugars	19.05	8.74	37.65	8.87	23.68	8.95	39.31	18.26
Rhamnose	1.20	1.02	1.88	0.93	1.27	1.28	2.23	1.73
Fucose	T	T	T	T	T	T	T	T
Ribose	T	T	T	T	T	T	T	T
Arabinose	11.58	5.92	30.31	3.62	9.37	4.11	27.08	6.30
Xylose	1.03	0.22	0.13	0.40	2.05	0.14	0.27	0.35
Mannose	0.67	0.26	0.30	0.15	1.01	0.21	0.21	0.12
Galactose	3.43	1.64	4.64	2.71	6.22	2.04	8.93	9.12
Glucose	1.14	0.68	0.69	1.06	3.19	1.36	0.77	0.60

T = traces

Table 3: Sugar composition of pectin fractions extracted from lime and orange fiber-pectin (g/100g pectin fraction).

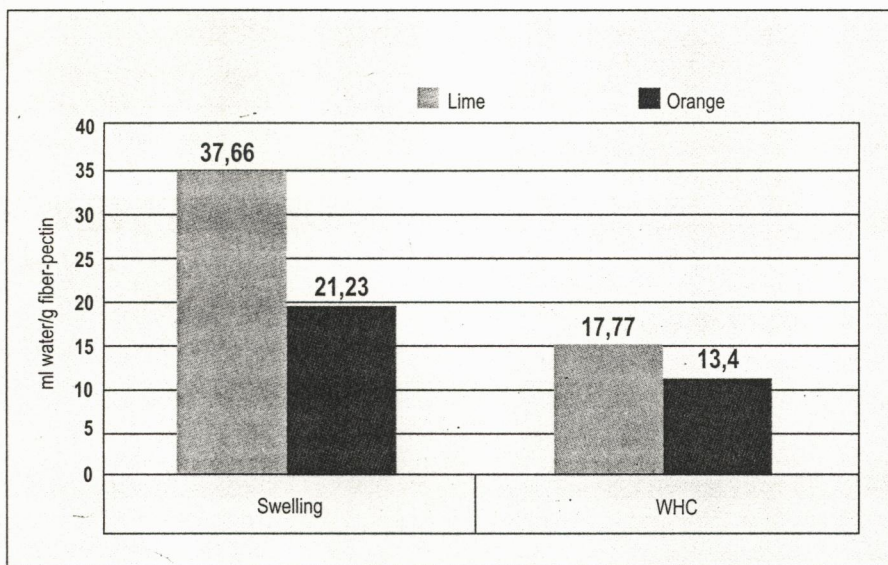


Fig. 2: Swelling and water-holding capacity (WHC) of lime and orange fiber-pectin.

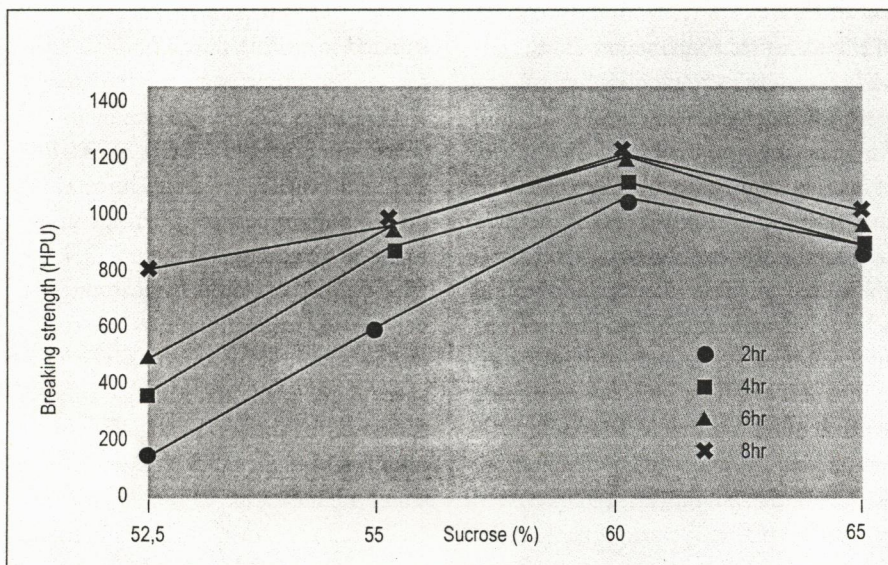


Fig. 3: Effect of sucrose concentration and holding time on breaking gel strength of lime fiber-pectin.

lime fiber-pectin was the lowest fraction (15.96%), whereas it represented 24.89% of the total pectin in orange fiber-pectin. This may explain the high calcium content found in orange fiber-pectin, since pectin molecules in the middle lamella are linked together by calcium bridges (JARVIS, 1984). In orange peel cell walls more pectin has obviously been rendered insoluble by the formation of calcium chelate complexes than in the lime peel cell walls. The sum of HSP and OHP fractions represented 45.52 and 58.59% of the total pectin in lime and orange fiber-pectin, respectively. Although these pectin fractions are firmly bound in the primary cell wall via acid- and alkali-labile bonds

(MASSIOT et al. 1992), a greater part could be solubilized from lime peels. This is probably due to different composition and distribution of neutral sugars in the pectin side-chains linked to hemicellulose and cellulose. Under acidic hydrolysis the glycosidic linkages of the furanosides are 10-1000 times more sensitive than the pyranosides (ASPINALL, 1982). Sugar composition of various pectin fractions obtained from lime and orange fiber-pectins (Table 3) showed that WSP of lime fiber-pectin contained higher AGA content (66.17%) than that from orange fiber-pectin (59.60%). A DM of 89.60% was observed in the WSP fraction from lime fiber-pectin.

The corresponding fraction from orange fiber-pectin had a DM of only 77.51%. Similar observation has been reported by RALET et al. (1994) who found DM of 91% in the acid-extracted pectin from extruded lemon peels. WSP of lime fiber-pectin was characterized by a low DAC compared to orange fiber-pectin. Moreover, total neutral sugar content of WSP from lime fiber-pectin was lower than that found in orange fiber-pectin. Compared to orange fiber-pectin arabinose, xylose and mannose were the only sugars found in higher concentration in WSP from lime. As for the water-insoluble pectin fractions (OXF, HSP and OHP) the OXF was characterized by the highest AGA content and lowest DAC and neutral sugar content compared to other pectin fractions. On the other hand, HSP had the highest DAC and neutral sugar content. Similar structural features of OXF and HSP have been observed in cherry fruits (SILIHA and GIERSCHNER, 1998) and in carrots (MASSIOT et al., 1992; SILIHA et al., 1996). Regarding the individual neutral sugars, the highest xylose and mannose content was present in the WSP fraction, while the highest arabinose and rhamnose content was found in the HSP fraction.

Functional properties

Swelling and hydration capacity of lime and orange fiber-pectins are presented in Fig. 2. Higher swelling and water-holding capacity were observed in lime than orange fiber-pectin. This is probably due to the extraction of more pectin encompassing the cellulose microfibrils, thus enhancing the hydration of cellulose. SHOMER et al. (1984) found that degradation of pectin by pectinase treatment of tomato juice enhanced swelling of the cellulose. Swelling and water-holding capacities reported in the present study exceeded those reported for wheat bran, pea hulls, sugar-beet fiber and carrot fiber (AUFFRET et al., 1994).

Gel formation is considered the most important criterion for fiber-pectin. In order to study optimum sugar concentration and holding time required for the

gel formation, gels were prepared with different sucrose concentration and kept for 2, 4, 6 and 8 hrs at 20°C before measuring. The breaking gel strength was expressed in HPU (Fig. 3). It is evident that the breaking gel strength increased with increasing sucrose concentration up to 60%. As expected for commercial citrus pectin the maximum breaking gel strength was at 65% sucrose concentration (results not shown). With increasing holding time there was gradual increase in breaking gel strength. Maximum breaking gel strength of lime fiber-pectin increased by 9.5% after 8 hrs holding time. Therefore, holding time of 2 hrs, which is the standard time for HPU determination, was used for further gel preparation.

The breaking strength of gels correlated with the amount of added gelling agent (Table 4). Orange fiber-pectin at 0.8% showed one fourth the breaking strength displayed by lime fiber-pectin. Utilization of orange fiber-pectin was tested in combination with lime fiber-pectin at different proportions. A mixture consisting of 3 parts lime fiber-pectin and one part orange fiber-pectin resulted in breaking gel strength comparable to lime fiber-pectin alone.

Although the breaking strength of gel prepared with 0.8% lime fiber-pectin was 60% of that found for gel of commercial citrus pectin, the AGA content of lime fiber-pectin is considerably low. Taking into account AGA content of 72% for commercial citrus pectin and 66.6% AGA in the WSP of lime fiber-pectin, which is only 38.52% of the total AGA, it is clear that the AGA content of lime fiber-pectin used for gel preparation represents only 20% of that from commercial pectin. Increasing the amount of lime fiber-pectin to 0.9% resulted in a breaking gel strength (2302 HPU) exceeding that of commercial citrus pectin (2272 HPU), thus indicating a great potential for utilization of lime fiber-pectin in food technology.

Strawberry jams containing lime and orange fiber-pectins were organoleptically evaluated for color, taste, texture and preference (Table 5). There were no significant differences in various

Type of gelling agents	Amount of gelling agent		
	0.4%	0.6%	0.8%
Commercial pectin	759	1475	2272
Lime fiber-pectin	495	916	1366
Orange fiber-pectin	72	188	344
Lime/orange fiber-pectin 3:1	450	839	1306
Lime/orange fiber-pectin 2:1	371	691	1122
Lime/orange fiber-pectin 1:1	259	586	939

Table 4: Breaking strength of gels prepared with commercial citrus pectin and fiber-pectin from lime and orange peels (as HPU).

Type of gelling agents	Color	Taste	Texture	Preference
Commercial pectin (0.5%)	3.0 ^b	3.0 ^{ab}	4.4 ^a	3.1 ^b
Lime fiber-pectin (0.5%)	4.2 ^a	3.7 ^a	3.7 ^{ab}	4.4 ^a
Orange fiber-pectin (1.0%)	3.3 ^b	2.4 ^{bc}	2.6 ^c	2.7 ^b
Commercial strawberry jam	4.5 ^a	3.1 ^{ab}	3.1 ^{bc}	4.4 ^a
L.S.D. at 0.05	0.79	0.88	0.80	0.89

L.S.D.: Least Significant Difference. Same letter means no significant difference.

Table 5: Sensory attributes of strawberry jams prepared with commercial pectin and fiber-pectin as compared to commercial jam.

sensorial properties tested between strawberry jam made by lime fiber-pectin and jam made by commercial citrus pectin or the commercial jam. In contrast jam made by orange fiber-pectin had inferior organoleptic attributes.

In conclusion, the extraction of fiber-pectin from lime peels proved to be a promising process, which is simple, inexpensive and yielding gels with breaking strength comparable to the commercial citrus pectin.

Acknowledgement

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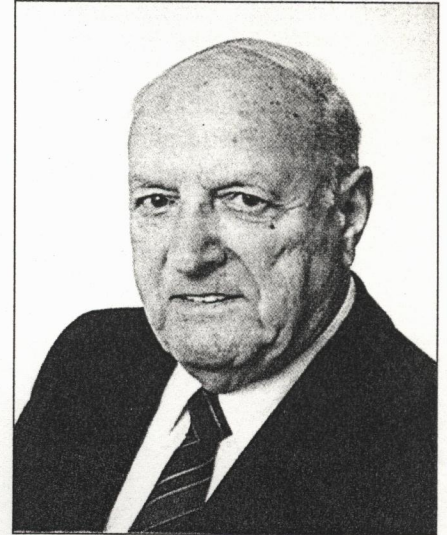
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Walter Hamester, 80 Jahre

Peter Schuhmann

Am 24. Januar 2000 vollendete Walter Hamester sein 80. Lebensjahr. Herr Hamester kam im Verlaufe seines überaus erfolgreichen Berufslebens früh zur Kartoffel, die ihn dann bis zum heutigen Tage nicht mehr verlassen sollte. Als gelernter Großhandelskaufmann war er in vielen Positionen des Kartoffelhandels und dessen Gremien, insbesondere im norddeutschen Raum tätig. Seine Verdienste betreffen aber auch die laufende Aktualisierung der Berliner Vereinbarungen und der RUCIP, den Aufbau des Gutachter- und Sachverständigenwesens, sowie dessen Umsetzung in der Praxis. Von 1949 bis 1985 war er in der Kartoffelverarbeitung der Raiffeisen HaGe Kiel tätig. Davon viele Jahre als deren Leiter. Seit über 40 Jahren wirkt er am Hamburger Großmarkt als Mitglied der Notierungskommission. Er war langjährig Mitglied und Vorsitzender des Verbandes der Kartoffelkaufleute Nord und ist heute Ehrenvorsitzender des Verbandes. Sein engagiertes Wirken im Dienste der deutschen und europäischen Kartoffelwirtschaft wurde im Jahre 1991 mit der Verleihung des Bundesverdienstkreuzes am Bande gewürdigt. Die Erfolge der alljährlich stattfindenden Hamburger Kartoffelbörse sind eng mit dem Namen Walter Hamester verknüpft. Die diesjährige Jubiläumsbörse (50!) nimmt Gelegenheit, seinen Anteil an diesem Ereignis gebührend hervorzuheben. In seiner Eigenschaft als Lehrsachverständiger hat er auf dem Gebiet des Kartoffelhandels hunderte von Nachwuchskräften ausgebildet. Letzteres gilt insbesondere auch für die neuen Bundesländer. Als Schiedsrichter



Walter Hamester

nach RUCIP und Berliner Vereinbarungen erwarb er sich große internationale Anerkennung. Einen festen Platz in der Kartoffelwirtschaft hat er sich als Autor bzw. Mitherausgeber solcher Fachbücher wie »Fachfragen für die Kartoffelwirtschaft« oder »Weltkatalog der Kartoffelsorten« erarbeitet. Hervorzuheben sind auch seine außerordentlichen Aktivitäten zur Versorgung der Stadt St. Petersburg mit Kartoffeln im Jahre 1990, sowie viele Gespräche und Treffen mit hochrangigen Persönlichkeiten aus Politik und Wirtschaft in Russland. Walter Hamester ist ein Mann der Tat. Was er denkt sagt er, was er sagt macht er, was er macht wird gut. Engagement, Erfahrung und Fachwissen, sowie Blick für das Wesentliche und das Neue sind Merkmale des Wirkens von Walter Hamester. Dazu kommt noch sein Talent im Umgang mit Menschen, welches Vertrauen schafft und Kräfte mobilisiert. Alle, die ihn schätzen und ihm viel zu verdanken haben, wünschen ihm weiterhin beste Gesundheit und persönliches Wohlergehen.