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Development of a Process for the Extraction of Pectin from Citrus Fruit Wastes viz. Lime peel, Spent guava extract, Apple pomace etc.

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Abstract

A method for determining the yield of pectin from various citrus fruit wastes viz. lime peel, spent guava extract, apple pomace etc. were outlined. The necessary steps in flow diagram showed the extraction procedure. Physical characteristics of different citrus fruit wastes were determined. Results showed that total solid, total undissolved solid and total dissolved solid calculated for fresh lime peel were 30.8%, 9.58% and 21.22% respectively. Similarly for spent guava extract the total solid, total undissolved solid and total dissolved solids were determined as 17%, 0.6%, and 16.4% and for fresh apple pomace 17%, 5.2% and 11.8% respectively. Effects of pH, temperature and time for the optimum pectin yield from those kinds of wastes are also studied. Results showed that optimum pH, temperature and time for pectin extraction from lime peel, spent guava extract and apple pomace were 1,80^{0}c, 30min; 5, 80^{0}c, 45min and 2, 70^{0}c, 15 min respectively. The optimum yield of powdered pectin under these optimized extraction conditions were found 0.05%, 0.06% and 0.05% for lime peel, spent guava extract and apple pomace respectively. So according to experimental studies spent guava extract was found to be most suitable for pectin extraction. Moreover the extracted pectin from different kinds of citrus fruit wastes could be utilized for formulation of different processed fruit products.

Key words: Extraction, pectin, citrus waste, pH, temperature, time

Introduction

The term pectin was first described and isolated by Henry Braconnot in 1825. Pectin consists of a linear backbone of randomly connected (1 fi 2) a-d-galacturonyl units partially esterified with methanol. The galacturonyl units are occasionally interrupted by (1 fi 2)-linked a-l-rhamnopyranosyl residues. The homogalacturonan sections are called 'smooth' and the rhamnogalacturonic regions are called 'hairy'. Neutral sugars are also present as side chains in different amounts depending on the pectin source and on the extraction method used (Kjoniksen et al., 2005). A valuable by-product that can be obtained from fruit wastes is pectin. Pectin exists in varying amounts in fruit cell walls and has important nutritional and technological properties, mainly because of its ability to form gels (Westerlund etal, 1991). Pectin is a polysaccharide having properties such as gelation and emulsion stabilization which make it useful in the manufacture of food, cosmetics, and medicine. It is a normal constituent of food and may therefore be safely ingested. Citrus peel, a by-product of the citrus processing industry, is a suitable source of pectin (Sakai and Okushima 1980).Pectin is a naturally occurring substance present in all plant tissue, calcium pectin being present between the cell walls and serving as a strengthening or building agent. Fruits naturally possessing relatively large amount of pectin include lemons, bitter oranges, apples, quinees, grooselevvies, currants and plums. It is less plentiful in fruits such as black berries, raspberries, strawberries and cherries. In the presence of fruit juice the gel will usually form when the concentration of sugar, acid and pectin are 68, 1 and 1 percent respectively. Pectin is usually extracted by placing the peel in vats of water, bringing the mixture to a boil as a slurry, and adding concentrated hydrochloric or sulfuric acid to adjust the pH to about 2.0.Filtration of the extract is a tedious process because the extract, containing pectin and disintegrated peel, is corrosive and viscous(Sakai and Okushima 1980). There are several ways to hydrolyze protopectin in citrus peel to water-soluble pectin and many of these methods have been reviewed by (Kertesz 1951) The commercial extraction of pectin is usually accomplished with mineral acids such as sulfurous (Joseph and Havighorst 1952, Wilson 1925) sulfuric (Snyder 1970)),

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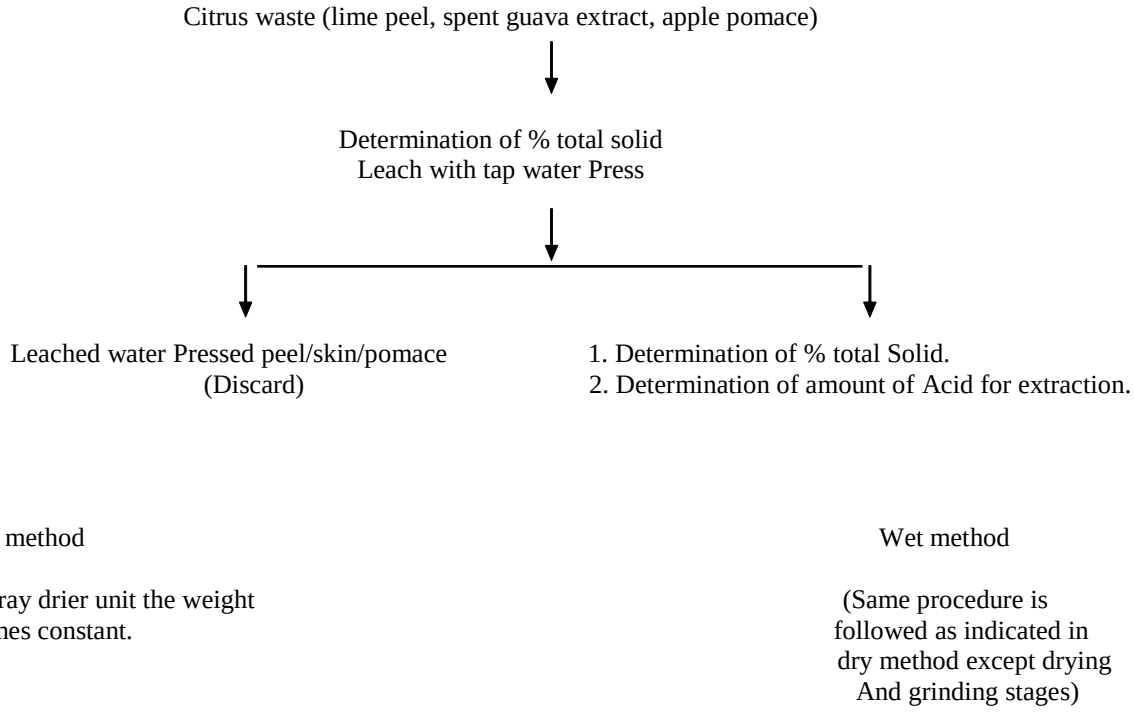
hydrochloric (Myers and Baker 1929), a combination of hydrochloric with ion-exchange resins (Doesburg 1973, Huang 1973, U S Patent 1973) or nitric acid (Rouse and Crandall 1976). Generally, extraction temperatures range from 80° to 100℃,time of extraction from 20 to 60 minutes and pH from 1.4to 2.6(Rouse and Crandall 1976). An extraction process is the most important operation to obtain pectin from vegetal tissue. Pectin extraction is a multiple-stage physical–chemical process in which hydrolysis and extraction of pectin macromolecules from plant tissue and their solubilisation take place under the influence of different factors, mainly temperature, pH and time (Kertesz 1951).Pectin extraction has been studied by several authors. (El-Nawawi & Shehata 1987) investigated the factors affecting the extraction of pectin from orange peel where the maximum yield was obtained using hydrochloric acid (90℃, pH 1.7 and 120 min). (Paga´n & Ibarz 1999) studied the extraction and the rheological properties of pectin from peach pomace, where the maximum yield was obtained using 70% nitric acid, 80℃, pH 1.2 and60 min. (Virk & Sogi 2004) studied pectin extraction and characterization from apple peel waste and revealed that citric acid was more effective than hydrochloric acid. Rehmann et al. 2004 extracted pectin from mango peels with sulfuric acid, and the maximum yield was obtained at 80℃ and pH 2.5 with an extraction time of 120 min. (Schemin et al. 2005) carried out a practical follow-up to pectin extraction from apple pomace and observed that the pectin yield was higher with 6.2 g per100 mL of citric acid and a reaction time around150 minutes. (Faravash & Ashtiani 2007) determined the effects of extraction time and pH variation on the yield of pectin isolation from pomace peento peaches, and the maximum yield of pectin was obtained at initial pH 2.5, EV of 1.5 and acid washing time of 120 min. The normal processes of ripening cause the dehydration of insoluble proto pectin into pectin's (or pectinic acids) and associated polysaccharides, and many of the physical changes in the structure of fruit and vegetable tissues coinciding with ripening are due to these changes in the pectin constituents. The pectin eventually passes into a gelatinous condition slightly soluble in water. High jelly grade pectin's are those where minimum chain dehydration has taken place and about half of the glacturonic acid groups are condensed as methyl esters. Pectin acid is the completely demethylated product possessing no power of forming sugar acid gels as required in the preserving industry. Citrus pectin is usually sold in a finely powdered condition. Pectin is available commercially in both liquid and powdered form. It has generally been extracted from either apples or citrus fruits. Pectin's are graded for the food industry. Grading is done according to sugar-carrying power. Pectin is partial methyl esters of polygalacturonic acid and their sodium, potassium, calcium and ammonium salts obtained by extraction in an aqueous medium of appropriate edible plant material, usually citrus fruits, apple & sunflower. In some type of pectin, a portion of the methyl esters may have been converted to primary amides by treatment with ammonia. Commercial product is normally diluted with sugars for standardization purpose. These are further specified to pH value, gel strength, viscosity, degree of esterification and setting characteristics. Commonly used pectin are of two different types mainly high methoxyl pectins (HM-pectin) having degree of esterification (D.E) more than 50 % and low methoxyl pectin (LM-pectins) having degree of esterification less than 50 %. High methoxyl pectin are further classified as ultra rapid set, rapid set, medium rapid set and slow set. Low methoxyl pectin are also specified as conventional and amided type. The degree of amidation (D.A.) indicates the presence of carboxyl groups in the amide form .The estimated annual worldwide production of pectin is 7,250 metric tons, approximately 60% of which is produced from citrus variety. California produces approximately 30% of the world supply (Crandall et al 1978). The citrus processing industry in Florida annually produces over 7, 79,000 metric tons of dried citrus peel, mainly orange and grapefruit for use as cattle food (Kesterson et al 1977). Currently none of this peel is being made into valuable product, pectin pomace, which would yield (20-30) % pectin (Rouse 1977). Thailand is the greatest exporter for canned pineapple, gained a lot of money increased in every year. In the year of 1996, 7100 million baht of money were gained (Ateeveerakul 1984). The pineapple processing industry has to get rid of waste like pineapple peel because of its deteriorating behavior. Pectin in apple pomace is mainly present in the form of protopectin, an acid soluble polysaccharide. The condition for the acid extraction of pectin is somewhat obscure in the literature and it is very difficult to visit processing units(Schemin et al 2005) The pectin has a ready market in India at all the time. It is assumed that total production of pectin in the country is about 70-75 tons per year, while the production is little compared consumption, which is estimated to be around 180 tons per year. During 1920,s and 1930,s fruit processing industries would build extracted pectin from dried apple pomace and citrus peel would be used to create delicious apple juice in both the United States and Europe. It is used not only be sold as a liquid extract but it is now offered as also in dry powder. These two forms are not interchangeable and as a result can be used in specific recipies. Liquid pectin is added to the cooked fruit and sugar mixture immediately after it is removed from the heat. Powdered pectin is mixed with the heated fruit juice or fruit pulp. Researchers are investigating the physiological benefits of consuming larger amounts of pectin in the diet (Braddock and Crandall 1978). If additional research substantiates that pectin significantly lowers serum cholesterol level in human then there may be a large increase in demand for citrus pectin and citrus waste pectin. The field of uses and applications of pectin show that there is vast consumption scope of pectin. It is very widely used in food and food processing industries. These are 4

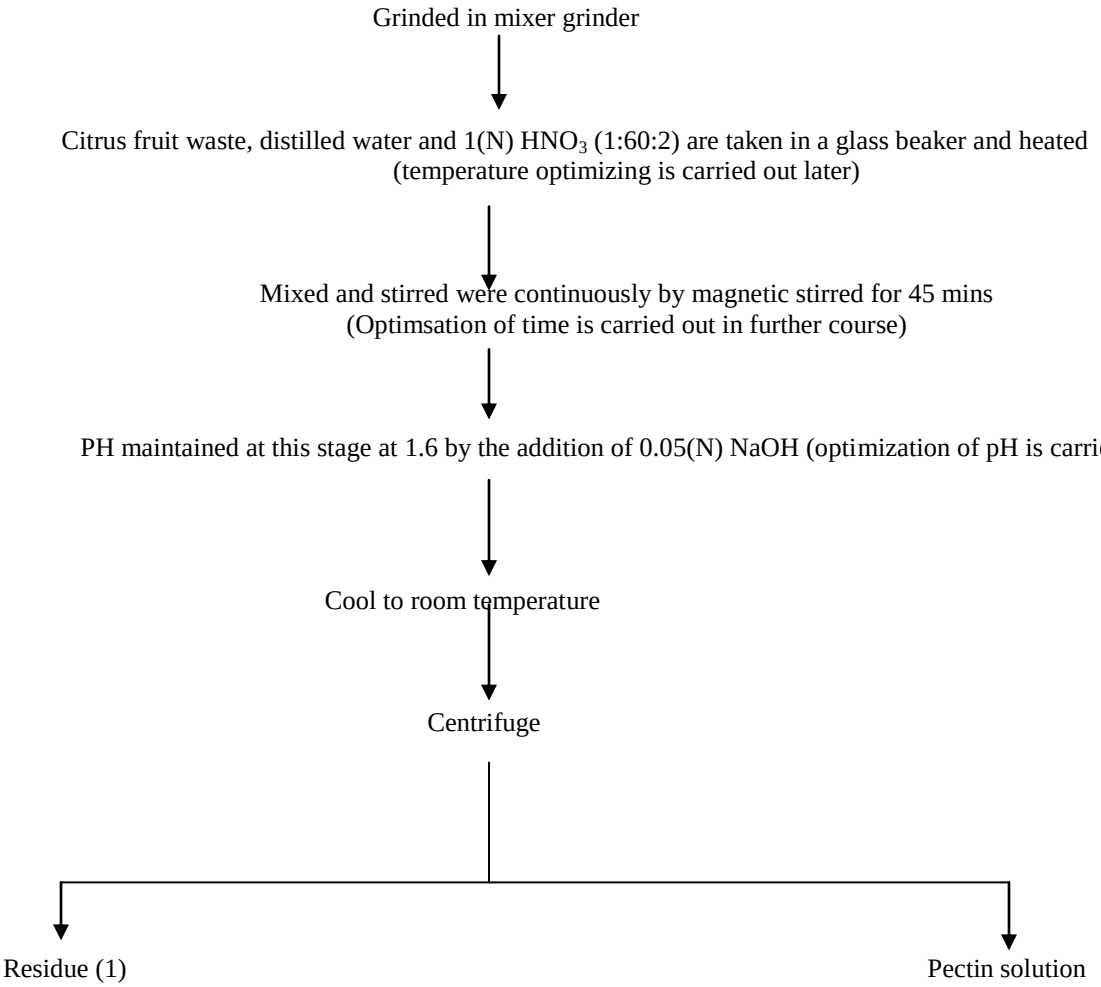
important ingredient and basic raw material for a large number of food products. For example, it is used in preparation of jam, jelly, sauces, pickles, ice cream, confectionary, drinks and a number of various food products. The market potential can be analyzed and a number of various food products. The market potential can be analyzed on the basis of the growth prospects of its users industries. The food processing units have been mushrooming at a rapid pace. Apart from the indigenous consumption, there is a demand of pectin in export market. This industry may prove to be a good foreign exchange earner. Fruit wastes, which are highly perishable and seasonal, is a problem to the processing industries and pollution monitoring agencies. Suitable methods have to be adopted to utilize them for the conversion into value-added products (Nand 1998). Now in the present study a method of extraction of pectin from citrus fruit wastes viz. lime peel, spent guava extract and apple pomace under different processing conditions viz. pH, temperature, time are evaluated to achieve potential economic value of pectin. Moreover it is also possible to extract pectin from other categories of citrus wastes viz. orange peel, pineapple peel, mango peel etc. to meet the growing demand of pectin in the current socioeconomic scenario.

Material and Methods

Fresh citrus wastes viz. lime peel, spent guava extract and apple pomace were purchased from the local food processors of Sodepur market, Kolkata, adjacent to the institute and taken as quickly as possible for experimental study. Chemicals e.g. concentrated HNO_3, NaOH and C_2H_5OH were used as laboratory grade reagent. Tap water was used in the leaching step and distilled water in the extraction stage. Some important mechanical devices as well as instruments viz. hot plate stirrer with thermostat control, cold centrifuge (Remi), vacuum filter and freezer were required to carry out efficient pectin recovery from citrus waste. Raw materials were prepared by the following way. Lime peel, spent guava extract and apple pomace are blanched with boiling water for 5 minutes and dried overnight at 60°c. Finally keep it in tightly closed container i.e. plastic bag till use. Now weighed citrus waste viz. lime peel, spent guava extract and apple pomace are added with distilled water and heated at 90°c for 45 minutes. During extraction with concentrated nitric acid solution usual level of pH is 1.6 and maintained by the addition of 0.05 (N) sodium hydroxide solutions. After this operation the solution attains to desired soluble solid content at 90°c followed by filtration. The concentrated solution is cooled to room temperature and centrifuged in a Remi cold centrifuge for 20 minutes at a speed of 12000 r.p.m and at -12°c. Recentrifugation is necessary with hot water at 60°c until dissolution of precipitate. 95 %(v/v) ethanol (1:1) is added to it under refrigerated condition and kept it for four hours for the development of precipitate. It is now centrifuged, dried at 40°c overnight and grinded. Finally the desired product pectin in crude form is kept in tightly sealed container or plastic bottle. The detailed flow sheet will be demonstrated by the following figure (Figure1).

Figure1. Nitric acid extraction of pectin from fresh citrus waste viz. lime peel, spent guava extract, apple pomace





Combine Pectin Solution (1) and (2)

1. Filter it by vacuum filter
2. Stirr it well into 95% ethanol (1:1) by volume
3. The precipitate stands for 4 hours under refrigerated condition
4. Centrifuge it and wash again with 95% ethanol solution
5. Dried in a Tray Drier at 40°c for overnight, grinded and kept in a tightly sealed bottle

Physical characteristics of raw materials

Determination of total solid (%). The moisture content of the sample viz. lime peel, spent guava extract and apple pomace were determined by conventional hot air oven technique under (100-105)°c temperature for (4-5)hours until constant weight comes. Now total solid is determined from it.

Determination of total undissolved solid (%). A finite quantity of citrus waste viz. lime peel, spent guava extract and apple pomace undergoes ashing operation under 550°c for (4-5) hours. The quantity of material left as ash gives directly the total undissolved solid (%).

Determination of total dissolved solid (%). The total dissolved solid (%) can be determined by deducting total undissolved solid (%) from total solid (%).

Optimization of process conditions for pectin extraction

Determination of optimum pH for pectin extraction. pH is optimized by varying it (1-6) for lime peel, (1.5-6) for spent guava extract and (1-6) for apple pomace at specified temperature of 90°c and time of 45mins respectively.

Determination of optimum time for pectin extraction. Optimization of time is carried out by varying it 15min, 30min, 45min and 60min respectively for each kind of citrus waste under constant pH and temperature.

Determination of optimum temperature for pectin extraction. Temperature optimization is carried out by varying it between (60-100) °c, every 10c interval for each category of citrus waste viz. lime peel, spent guava extract and apple pomace under optimum pH and optimum time.

Results and Discussion

It is important to start the analytical work with a known quantity of fresh citrus waste because this will be used in calculating the yield of pectin on a fresh citrus waste basis. The percentage of total solids found in the fresh citrus waste indicate how much soluble solids, pectin and other components besides water are available in the lime peel, spent guava extract and apple pomace. After the fresh peel, spent extract and pomace has been leached, pressed and total solids determined then the quantity of total undissolved solid can be determined by measuring ash content. Finally the amount of total dissolved solids removed by leaching can be calculated as a difference. The total solid, total undissolved solid and total dissolved solid calculated for fresh lime peel are 30.8%,9.58% and 21.22% respectively. Similarly for fresh spent guava extract are 17%, 0.6%, 16.4% and for fresh apple pomace are 17%, 5.2% and11.8% respectively. The more soluble solids that are leached from the fresh citrus waste the greater will be the yield of pectin from a given weight of fresh waste. The large amount of soluble solids leached from the fresh citrus waste will create a greater load on the waste treatment facility (Rouse and Crandall 1976).

Extraction of pectin by mineral acid. A good number of mineral acids can be used to extract the pectin from the other cellular material. For the determination of acid requirement, a small amount of fresh citrus waste, water and acid are heated together. The pH level maintained at this stage is generally1.6 (Kertesz 1951). Here approximately sixty times as much 0as fresh waste viz. lime peel, spent guava extract and apple pomace are heated together with the acid. Now for the extraction of pectin 25gm of dry solid from leached peel, spent extract or pomace are normally extracted at one time. The optimum pH, temperature and time for pectin extraction as well as yield are determined.

Effect of pH on the pectin yield (%) from lime peel, spent guava extract and apple pomace: Different pH is tested for the extraction of pectin from fresh lime peel, spent guava extract and apple pomace. The optimum pH is determined by considering 20ml of the extract where temperature and time are maintained constant at 90°c and 45min. The results are shown in figure-2. Results showed that at pH 1, 5 and 2 the pectin yield is maximum viz.0.05%, 0.06% and 0.05% for lime peel, spent guava extract and apple pomace respectively. So these pH levels are considered to be optimum for this study. Moreover spent guava extract is most suitable citrus waste for utilization for this study.

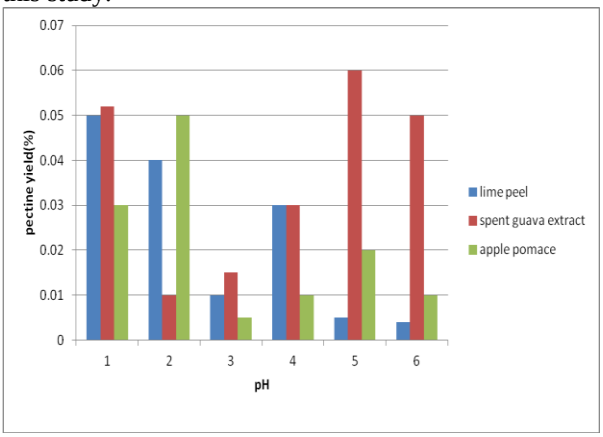


Figure 2. The yield of pectin (%) vs. pH from lime peel, spent guava extract and apple pomace

Effect of temperature on the pectin yield from lime peel, spent guava extract and apple pomace. The effect of temperature on pectin yield is studied by carrying out the experiment at variable temperatures. The optimum temperature is determined by considering 20ml of the extract at constant pH and time viz. 1 and 45min respectively. The results are shown in figure-3. It is evident from the result that the maximum pectin extraction (%) e.g. 0.06%,0.09% and0.06% is obtained at 80°c ,80°c and 70°c temperature for lime peel, spent guava extract and apple pomace respectively. So these temperatures are considered as optimum in this study. It is also being observed from the

datas that spent guava extract is found suitable citrus waste for pectin extraction.

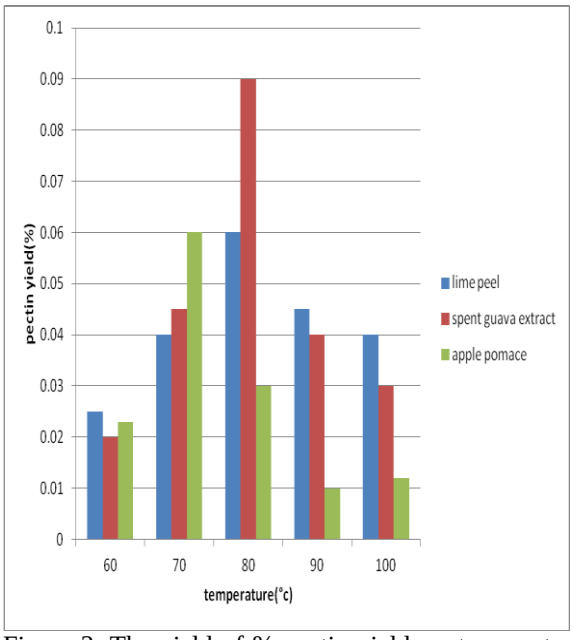


Figure 3. The yield of % pectin yield vs. temperature from lime peel, spent guava extract and apple pomace

Effect of time on the pectin yield from lime peel, spent guava extract and apple pomace. Pectin extraction is carried out by varying time. In this case the optimum time for pectin extraction is determined by considering 20ml extract volume. The results are shown in figure-4. Results showed that optimum pectin yield (%) viz.0.055%, 0.06% and 0.02% is achieved at extraction time of 30min, 45min and15min for lime peel, spent guava extract and apple pomace respectively. So these times are considered as optimum time for pectin extraction in this study. Now it is evident from the result that maximum pectin extraction is obtained in case of spent guava extract.

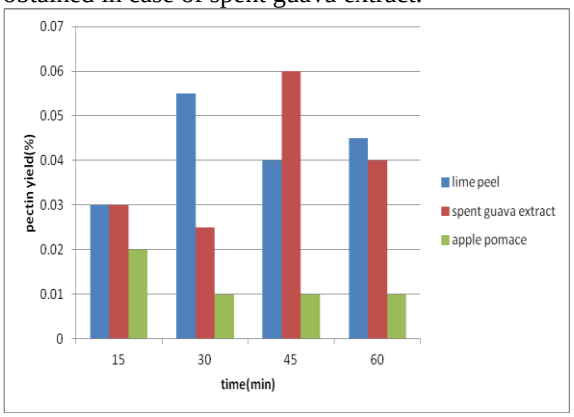


Figure 4. The yield of pectin (%) vs. time from lime peel, spent guava extract and apple pomace

So in this work a process is developed for the extraction of pectin from different kind of citrus fruit wastes viz. lime peel, spent guava extract and apple pomace. The process development is carried out by standardizing various process conditions viz. pH, temperature and time respectively. It is being observed that maximum pectin yield is obtained (0.09%) for spent guava extract at 80°c as compared with lime peel and apple pomace. By following the same way the work can be continued for other citrus wastes e.g. mango peel, pine apple waste, orange peel, water melon rind and seed etc. and an effective comparative study can be carried out in terms of pectin extraction (%) with respect to waste utilization process. Finally the extracted pectin is used to formulate various types of processed fruit products and others e.g. jelly, jam, marmalade, synthetic jelly, thickener and stabilizer etc.

Pectin recovery. Now the efficacy of extraction of pectin from various citrus fruit wastes viz. lime peel, spent guava extract and apple pomace are controlled under optimum values of pH, temperature and time respectively. The pectin solution is centrifuged under high speed cold centrifuge (12000r.p.m and-12°c) and washed for complete extraction. The pectin solution is concentrated and precipitated with 95% ethanol solution (1:1) by volume. Water and alcohol washes are used to remove any residual acid entrapped in the pectin precipitate. The pectin is dried in a batch tray drier at 40°c for overnight, quickly grinded and stored in a sealed plastic bottle under refrigerated condition.

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