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Encapsulation Techniques of Nutraceutical Extracts and Factors Influencing the Processes - A Review

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Abstract : Plant extracts contain numerous functional and nutritional components such as polyphenols, antioxidants, tannins, anthocyanins, ascorbic acid, folic acid, and other vitamins. These compounds when delivered into our gut might result in anti-inflammatory, anti-angiogenic, anti-infective, anti-ailment, anti-proliferative, anticancer, and antimicrobial functions. To preserve these components, the spray drying technique has been adopted as the most rational approach for heuristic processing and storage. Unfortunately, all these compounds are very much delicate since they are thermodegradative, photodegradative, and highly prone to oxidative damage during processing and long-term storage. Hence to protect their functionality, certain food-grade biopolymers like gelatin, gum arabic, maltodextrin, whey protein isolate, arrowroot starch, chitosan, and alginate are incorporated with them. These polymers embed those bioactive components and preserve their functional aspects during spray drying. They also aid in the controlled release of the encapsulated core compounds while they are passing down the digestive tract hence cater the target compounds.

Index terms **- Microencapsulation, biopolymer, bioactives, spray-drying, vitamin.**

I. INTRODUCTION

In recent years, diverse challenges are been faced by the industries to package the food bioactive compounds into the gut due to their interactive binding capabilities with proximate compounds and attenuated antimicrobial capabilities of complex coating matrices. Polyphenolics like epigallocatechin gallate, epicatechin gallate, resveratrol, luteolin, and curcumin have a wide range of anticancer and chemopreventive properties. Polymeric tannins like tannic acid, theaflavin, procyanidin B, and thearubigins have antimicrobial and antioxidant activities. Phytochemicals can induce apoptosis of tumor cells. Their functional aspects possess a very low half-life. Nanocarrier delivery system has led to protection, enhanced payload capacity, surface functionalization, non-antigenicity, biodegradability, high nutritional value, renewability, unique binding capability with nutrients, and steric hindrance towards drugs hence generally recognized as safe. Gelatin has been such a widely accepted nanopolymer with biocompatibility, low immunogenicity, actively modifying capability with target compounds, and is applicable for nutrient delivery (Ali et al, 2019).

Certain berries like Chokeberry extract have been rich in proanthocyanidins and polyphenols which are potent antioxidants but highly degradable. Hence encapsulation by maltodextrin and gum arabic has been providing the best shelf life extension for these bioactive. Here spray drying has been one of the best technologies standing out to be a one-stop solution to meet this requirement (Cujic, 2019).

The polyphenols, anthocyanins, and tannins of blackberries have been high in anti-cancer activities. Spray drying has been the most effective way of protecting these bioactives for a longer shelf life period. But these compounds are been heat-sensitive hence there have been quality losses of them. Thus encapsulation has been promoting the controlled release of these bioactive components. Among widely accepted coating agents, gum arabic and arrowroot starch are been used for this purpose. Their mixture has been proved to be tasteless, odorless, and non-toxic coating material. Optimizing inlet and outlet temperatures have been providing the best outcome for the product (Noguira et al, 2020).

Ascorbic acid has been a potent antioxidant and vitamin supplement but is highly degradable at a higher temperature, oxygen, and metallic ions. Hence encapsulating it with rice starch and gum arabic kind of substances has been protecting its viability and stability preventing undesirable and premature changes (Trinidade and Grosso, 2020).

Several environmental factors like pH, temperature, light, oxygen, moisture, and UV rays have been leading to ascorbic acid degradation which changes its color to yellow thereby resulting in unwanted reactions of it with other food ingredients. Therefore encapsulation of it has been preventing all these unprecedented adversities (Uddin et al, 2001).

The extracts of herbal tea (Orthosiphon stamineus) have been high in medicinal compounds like terpenoids, sterols, and polyphenols that have been antiangiogenic, antiproliferative, and antidiabetic, and diuretic. They have been acting against jaundice, inflammation, eruptive fever, edema, hepatitis, rheumatism, influenza, hypertension, and diabetes. Hence these compounds are been needed to be powdered to store them for a longer-term. Here spray drying has been used as the best option for having a powdered product for convenient consumption. The high temperatures of drying have been affecting the nutritional, functional, and bioactive properties of the product. Thus microencapsulation has been the best choice for preventing the thermal degradation of these bioactive compounds. Here whey protein isolate and maltodextrin are been best for the purpose (Pang et al, 2014).

Cocoa beans are been high in polyphenolic and antioxidant contents. However, the processing and storage of these have been resulting in a negative effect on degrading their volatile components. Thus microencapsulation by spray drying has been protecting these heat-sensitive bioactive components. Here whey protein isolate has been an excellent wall material that provides a barrier to the nutritional core components against oxidants and deteriorating agents within the oil-in-water interface of emulsion. This crust has been providing thermo-protection for the bioactive volatile compounds during processing and storage. The inlet drying temperature at 150°C has been providing maximum retention of polyphenols and volatile components with whey protein as coating material (Calva et al, 2019).

Whey protein isolates are been converted into gels at room temperature. These gels can form cold-set gels that preserve the sensitive bioactive compounds by coating around them which does not require further heat treatment. These have been extremely stable at varieties of pH and temperatures. These gels are been resistant to enzymatic degradation in the gastrointestinal tract. Hereby food-grade whey protein gel has been prepared with lipid loads. A functional compound called riboflavin has been immersed within this emulsion gel and converted to cold set gels which are been further analyzed for their digestibility and release of bioactive components. These cold-set gels are been prepared by incorporating the denatured whey protein isolate into calcium chloride solution dropwise to produce microgels (Egan et al, 2013).

Alginate a biopolymer isolated from brown seaweeds namely laminaria digitate and Laminaria Hyperborea has a structural analogy to the extracellular polysaccharides. It has been one of the less toxic and highly pure biopolymers available in nature. It has been resistant to acidic pH, easily soluble, and has been providing controlled release of functional components acting as a microencapsulating agent. It is been used to coat L-ascorbic acid via the spray drying technique. Thus alginate is been used as potent wall material for ascorbic acid encapsulation with high efficiency and yield (Marcela, 2016).

Wastes collected from wine industries are been rich in polyphenols, vitamins, and antioxidants which have high anti-bacterial, anti-inflammatory, and antioxidant properties. These compounds are highly thermo-degradable and photo-degradable. Thus their controlled delivery is been very necessary into the human gut for proper absorption. Hence microencapsulation of these functional components has been protecting them from undesirable damages and enhancing their stability as well as bioavailability. Alginate and chitosan mixture is been considered one of the possible coating materials to immobilize the polyphenolic compounds and impart appropriate medicinal aspects (Moschona and Liakopoulou, 2018).

Anthocyanins found in fruits and vegetables are been high in anti-cancer, antioxidant, and anti-inflammatory properties. Anthocyanins are been vulnerable to temperature, pH, oxygen, and light. Thus encapsulation of the anthocyanins has been helping protect them against all these degradations. So biological polymer has been the best choice for encapsulation. Among the available biopolymers, maltodextrin is been the best coating agent. These food-grade wall materials are been extremely stable and abundant. One such anthocyanin protecting agent has been cinnamic acid extracted from cinnamon which blocks oxidative damage and photodegradation for the stability of anthocyanins. Hence to enhance the efficiency of wall material, maltodextrin is been grafted with cinnamic acid to favor promising results of antioxidant, thermostable, and UV-protective activities of core functional components of purple sweet potato (Yi et al, 2016).

Folic acid, a member of the vitamin B family has a wide range of physicochemical activities in the human body. The folate compounds supplemented in foods are been highly sensitive to oxidative damage, photodegradation, thermodegradation, and pH during food processing. Thus protecting the stability and bioavailability of these compounds by encapsulating them with a semipermeable polymer matrix has been of prime importance. This has been providing controlled release of folates into the gastrointestinal tract and active absorption. Two suitable compounds alginate and pectin are been cross-linked with each other to provide a stable coating matrix for embedding folate (Madziva et al, 2005).

II. MATERIALS AND METHODOLOGIES

2.1. Materials

2.1.1. Raw Materials: Animal tissues, chokeberries, blackberries, gelatin, alginate, maltodextrin, gum arabic, arrowroot starch, ascorbic acid, rice starch, sinensetin, eupatorin, rosmarinic acid, whey protein isolate, curcumin, *Orthosiphon stamineus*, Cocoa beans, riboflavin, soybean oil, alginic acid with sodium salt, chitosan, grape skins, purple sweet potato, cinnamic acid, folic acid, pectin with potassium salt. All these have been collected from supermarket.

2.1.2. Chemicals: Deionized water, HCl, phenol, acetone, glutaraldehyde, carboxymethyl cellulose, polystyrene sulfonate, polyallylamine hydrochloride, span 20, cyclohexane, folin-ciocalteau reagent, Ferric Radical Antioxidant powder (FRAP), 1,1-diphenyl-2-picryl-hydrazyl (DPPH), tris-HCl, barium chloride, liquid paraffin, hexane, carnauba wax, polyisobutylene, ethyl cellulose, triethyl citrate, methanol, ethanol, 2,2'-azinobis-3-ethylbenzothiazoline-6-sulfonic acid (ABTS), acetonitrile, trifluoroacetic acid, gallic acid standard, epicatechin standard, petroleum ether, calcium chloride, sodium azide, thionyl

chloride, acetic acid, potassium chloride, sodium acetate. All these are bought from Sigma Aldrich, Loba Chemie, Emplura, Emsure, Merck, and Spectrochem.

2.1.3. Instruments and Apparatus: Spray dryer, ultrasonicator, desiccator, water bath, Whatman filter paper, microfluidizer, High-pressure liquid chromatography (HPLC), Ultra performance liquid chromatography (UPLC), sonicator, ESI-MS Electrospray ionization mass spectrometry (ESI-MS), magnetic stirrer, freeze drier.

2.2. Methodologies

Gelatin has been acquired in the form of granules or powder. It is been extracted via acid or alkali hydrolysis of animal tissues. Enzymatic hydrolysis is been done by matrix metalloproteinases. Cationic gelatin and anionic alginate interact with each other and form a polyelectrolyte complex which aids in vitamin delivery. Double protective nanocomplexes are been prepared by encapsulating the gelatin particle within hydrogel via chemical bonding, physical entrapment, and cross-linking. Self-assembly technique like hydrogen bond formation, hydrophobic interactions, and Π-Π stacking interactions between fish scale gelatin hydrolysate (FSGH) and plant phytonutrients forms nanocomplexes. FSGH is been obtained by hydrolyzing the fish scales with 6M HCl, 1% phenol(v/v) at 110°C. Then the FSGH is been dispersed in deionized water and flushed with phytonutrients by continuous stirring which results in nanoparticle formation finally. Nanoemulsification is been done by dissolving gelatin/lecithin complex with thymol solution at 15000 rpm for 2 mins in a microprocessor homogenizer. The Desolvation technique by alcohol/acetone has been allowed to dehydrate the gelatin particle and coil it up for hardening conformation. At 37°C and pH 7.0 curcumin-loaded gelatin nanocomplexes are been prepared. Besides for low molecular weight gelatin nano complex preparation, at first, the gelatin is been dissolved into acetone which precipitates high molecular weight (HMW) gelatin. After discarding supernatant containing low molecular weight acetone the rest of the HMW gelatin is been redissolved at pH 12 by dropwise addition of acetone for 12 mins. After stirring the solution for 10 mins glutaraldehyde as a cross-linker is been added and the resulting mixture is been stirred at 50°C for 3 hrs. Then 3 fold centrifugation has been done followed by redispersion of HMW gelatin particles precipitate with 30%v/v acetone and again subjected to centrifugation. Again the precipitate is been redispersed into the water to obtain purified gelatin nanocomplexes evaporating the residual acetone. Coacervation has been a technique where liquid-liquid separation with a heavier phase below and a lighter phase above is been done. Span 80 along with reservatrol is been added into gelatin aqueous phase heating at 40°C to give rise to nanoparticles. Then glutaraldehyde is been added finally to cross-link the delivery agent. On the other hand layer by layer coating of gelatin capsules is been done by incorporating carboxymethyl cellulose, polystyrene sulfonate, and polyallylamine hydrochloride at pH 6.0 in an aqueous solution of gelatin nanoparticle with EGCG. Each layer is been adsorbed for 30 mins then washed with deionized water and another layer is been added. Aqueous biopolymer of alginic aldehyde gelatin solution has been prepared by dissolving 10% alginate aldehyde with 10% gelatin and adding into the lipophilic solvent of cyclohexane in presence of a surfactant span 20 for getting a homogenous emulsion. Ultrasonication followed by high-speed stirring for 5 mins is been done to disperse the nanogels homogenously. Finally, they are been purified by centrifugation and washing (Ali et al, 2019).

Chokeberries are been collected from organic farming and segregated into two portions, one is frozen, thawed, and smashed to decant the juice, then the pomace is been considered as chokeberry waste extract(CWE). The other portion is been dried, smashed, and decanted, then the juice is been extracted with alcohol to retain the polyphenols. After that the polyphenols are been concentrated by evaporating the alcohol via a rotary vacuum evaporator and this extract is been considered chokeberry fruit extract(CFE). Maltodextrin and skimmed milk are been added to the CFE and CWE in a 20% (w/v) ratio and the solutions are been heated for 50°C for 12 hrs. followed by homogenization. Then the solutions are been spray-dried to obtain the globules. Total polyphenols content is been estimated via folin-ciocalteau reagent indirect method so also total anthocyanins content is been determined via Europian Pharmacopia 8.0. Their release rate is been calculated by comparing initial and final content analysis. The encapsulation efficiency(EE%) is calculated via the ratio of E/Etotal × 100, where E is a polyphenol, anthocyanin content in encapsulated beads and E total is the said in initial feed extract (Cujic, 2019).

Blackberries are been collected from organic farming and refrigerated for 24 hrs at 8°C. Then they are been thawed and ground to collect the pulp which is been sieved away from seeds and homogenized, then packed in polypropylene bags and enclosed within aluminum foils against photodegradation. Then they are been stored at -40 ± 3°C till drying. The ascorbic acid and anthocyanin content are been found to be 124.54±17.37 mg/100g solids and 114.71±8.50 mg/100g solids respectively. The coating agents like arrowroot starch has been containing ascorbic acid 45.05 ± 7.67 mg/100g solids and gum arabic has been containing the said about 42.47 ± 7.22mg/100g solids. The inlet temperature of the spray drier has been about 100-150°C and the arrowroot/gum arabic are been mixed in a 1:1 (w/w) ratio. The encapsulating agent and blackberry pulp concentration ratio have been about 0.5:1 to 2:1 (w/w). Their mixture has been homogenized for 5 mins and dissolved in water in a 1:2 (w/w) ratio. Spray drying is been done and the final powder is been stored in a polypropylene bag. The total ascorbic acid content is been measured by the folin-ciocalteau method, and the retention is estimated by the ratio of the ascorbic acid content of powder to the mixture. Total anthocyanin content is been measured by the acetone/Tris-HCl method and retention is been calculated by the total anthocyanin of powder and that mixture ratio. The antioxidant content is been measured by FRAP and DPPH assay (Noguira et al, 2020).

Rice starch is been extracted from rice flour by washing it with NaOH and distilled water. Then it is been heated at 50°C in an incubator with hot air sparging. Finally the resultant is been sifted in 0.35mm mesh to refine it. 10% ascorbic acid is been dissolved with rice starch in (w/w) ratio which is been kept in suspension with binding agent gelatin in (1:2% w/w ratio of gelatin: rice starch). The mixture is been heated at 45°C, stirred and homogenized, and finally spray dried at 120°C inlet and 92°C outlet temperatures. Besides 30% ascorbic acid is been dissolved with gum arabic and homogenized and stirred at room

temperature, finally spray-dried at 150°C inlet and 88°C outlet temperatures. All the samples are been finally stored in dark bottles to prevent photodegradation. Ascorbic acid stability is been measured by AOAC 1984 method. 0.5-1 g of sample is been extracted with 50 ml extraction solution with continuous agitation of 10 mins to release ascorbic acid. The resultant is been stored at 21°C at 60-65% relative humidity and analysis is been carried out at an intermittent number of 30 days intervals from 0-90 days. Samples stored at 45°C for 18 days exhibited relative humidity of 90% with a supersaturated solution of barium chloride, which are been analyzed in 0, 4, 11, 18, 23, 30, 37 days consecutive intervals. Now after spray drying the ascorbic acid retention is been considered 100% to calculate stability (Trinidade and Grosso, 2020).

300 ml of cyclohexane solution is been mixed with 0-6% of coacervate polyisobutylene in a round-bottomed flask. Now 6 g ethylcellulose is been added into the mixture at 25°C with continuous stirring at 1000 rpm. The mixture is been heated at 80°C for homogenizing followed by 18 g ascorbic acid particle addition with continuous stirring. When the mixture is been cooled down to 40°C for 1 hr, it is cooled to 25°C finally. The microcapsules are been finally decanted, hexane washed, and air-dried at room temperature. Besides 2 g ethyl cellulose is been dissolved into 10 ml alcohol at 40°C in a water bath. 1 ml triethyl citrate is been added to this polymer solution followed by the incorporation of ascorbic acid sonicated at 70 kHz to form a homogenous mixture. 210 ml of liquid paraffin and hexane in a 2:1 ratio is been mixed to form a continuous medium. Then the ascorbic acid polymer suspension is been transferred to this continuous medium to form an emulsion which is stirred at 1000 rpm at 28-55°C for 4 hrs. Then the microcapsules of ascorbic acid are been decanted, washed with hexane, and air-dried at room temperature. On the other hand, molten carnauba wax is been mixed with ascorbic acid and formed an emulsion. Then this emulsion is been diluted and agitated for 20 s, cooled rapidly in ice-cold water to form microparticles. These microparticles are then been decanted, washed, vacuum dried, and sealed within vials at room temperature. All these polymeric solutions of ascorbic acid previously homogenized are now been spray-dried with an inlet air of 200-300°C and outlet air of 70-95°C. Then these dried microcapsules are been obtained from the dryer and stored within a laboratory desiccator (Uddin et al, 2001).

Fresh Orthosiphon stamineus leaves are been washed with deionized water and dried at 37°C for 3 days and then ground. This dried powder is then been stored in air tight pouch within the desiccator. Then this powder is been ultrasonicated at 50°C for 90 min at 45 kHz to release the polyphenols. Then 50 % methanol is been added to this ultrasonicated powder to extract the compounds like sinensetin, eupatorin, and rosmarinic acid. This supernatant is been decanted by Whatman filter paper and the filtrate is been dried at 40°C to evaporate the excess methanol to obtain the solid concentrate. Now this concentrate is been diluted in 1.05% (w/v) proportion in 50 ml water and mixed with a solution of encapsulating agent containing 0.1%, 1%, 10%, and 30% (w/v) of maltodextrin or whey protein isolate. Then this entire solution is been magnetically stirred for 30 min at 40°C to obtain a homogenous mixture. Now 0.05%, 0.53%, 5.33%, and 10.67% of either whey protein isolate or maltodextrin-containing feed concentrate is been spray-dried at 180°C. Now 60% methanolic solutions of these dried powders are been prepared. Again about 10 mg/ml, 10 mg/ml, and 5 mg/ml of rosmarinic acid, eupatorin, and sinensetin stock solutions are been prepared respectively. Then these 3 are been diluted to form a 0.08 ug/ml concentration. Determination of the extracts is been done by the UPLC method having a mobile phase with solvent A as water: trifluoroacetic acid (20:0.001 v/v) and solvent B as acetonitrile: trifluoroacetic acid (20:0.001 v/v) at 24°C (Pang et al, 2014).

An oil in water nanoemulsion is been prepared by dissolving cocoa liquor and whey protein concentrate in a 1:3 ratio(w/w) in distilled water, then homogenized by a microfluidizer and preserved in an ice bath to protect against thermal degradation. This nanoemulsion is then been spray-dried at an inlet and outlet of 120°C and 69°C respectively. 0.1 g of this sample is then been defatted twice using petroleum ether and air-dried to remove the residual organic solvent. The catechins and methylxanthines are been extracted with 70% methanol with 0.1% trifluoroacetic acid. Then this mixture is been centrifuged at 3500 rpm for 10 mins and the supernatant is been decanted and filtered. The polyphenolic content is been measured by the folin-ciocalteau method using catechin and gallic acid standards per gram of defatted dry sample. The microencapsulation yield is been calculated in the ratio of polyphenolic contents in the final spray-dried powder to that of the initial emulsion. HPLC analysis is been done for methylxanthine and catechin with 0.1% trifluoroacetic acid as solvent A with acetonitrile and 0.1% trifluoroacetic acid as solvent B. The antioxidant capacity is been measured by DPPH and ABTS radical scavenging activity. Solid-phase microextraction is been done to extract volatile components. 2 g cocoa liquor or microcapsule powder mixed with distilled water is been transferred to a headspace vial and sealed, then agitated for 15 mins at 60°C to extract the volatile compounds. Then once when the fibre gets exposed in the vial, it is been agitated constantly for 30 mins at 60°C. Then this fibre is been retracted and inserted into the gas chromatographic inlet for analyzing the volatile components. The antioxidants, polyphenols, and volatile contents are been measured at successive temperatures of 4°C, 25°C, and 60°C with intermittent storage of 30 days. The retention value is been calculated by comparing the initial value of these functional compounds within the emulsion stage and the final value of them in microcapsule powder (Calva et al, 2019).

10% (w/w) whey protein isolate with 0.2% sodium azide is been prepared into deionized water at 40°C. This solution is been stirred overnight at ambient temperature for producing a homogenous mixture and hydration of protein matrices and swelling them. Then this solution is been heated in a water bath at 80°C at sustained temperature for 30 mins and rapidly cooled on ice for bringing to ambient temperature for further analysis. Then this denatured whey protein solution is been mixed with 20% (w/w) soybean oil emulsion and the entire mixture is been homogenized for 30 s. 0.05g/l equivalent riboflavin is been added to this denatured whey protein emulsion to prepare cold set microgels. These riboflavin-containing microgels are been tested for release of riboflavin over time for both enzyme-free and enzyme-containing gastrointestinal fluid. Centrifugation is been done and riboflavin content in the fluid is estimated spectrophotometrically, it is found that the enzymes do not affect the riboflavin content. The entrapment efficiency of riboflavin is been given by the ratio of the quantity of riboflavin initially loaded into the microgel and that finally released from the microgel (Egan et al, 2013).

The alginic acid with sodium salt is been dissolved in distilled water in a 3.5% (w/w) ratio and refrigerated for 24 hrs to obtain a stable viscous emulsion. Now ascorbic acid is been mixed with the alginate solution in 1:3.5, 2:3.5, and 2.5:3.5 ratios

and agitated at 1200 rpm for 10-15 mins at ambient temperature. The encapsulation yield is been expressed in the ratio of the weight of spray-dried powder collected to the original weight of all the components in the emulsion prepared for drying. The encapsulation efficiency is been given as the ratio of total ascorbic acid minus surface ascorbic acid content to the total ascorbic acid content expressed as a percentage. This surface ascorbic acid content is been measured by dissolving the capsules in 30 ml distilled water and analyzing the supernatant at 264 nm. The total ascorbic acid is been measured by dissolving the capsules in 10 ml ethanol and centrifuging the solution at 3000 rpm to analyze the supernatant at 245 nm. The encapsulation efficiency is been determined 30 days post-production process (Marcela, 2016).

The grape skins are been sonicated with ethanol in a sonicator at 35°C for 40 mins at pH 2.0, then centrifuged at 4500 rpm for 10 mins and the supernatant is been frozen at -20°C. Total polyphenolic content is been estimated by the folin-ciocalteau method and phenolic compounds are been identified by HPLC and ESI-MS. The HPLC is been done in a C-18 reverse phase column with 2% (v/v) acetic acid as solvent A and 100% acetonitrile as solvent B in ratios of 85:15, 60:40, and 45:55 respectively. The ESI is been performed at 300°C capillary heating and a 3.7-kilovolt ionization source. The antioxidant activity is been measured by DPPH solution. Sodium alginate 1% (w/v) is been prepared with water. Chitosan about 3% (w/v) is been prepared into 1.5% (w/v) calcium chloride solution and homogenized by magnetic stirrer. Now the polyphenolic extracts are been added to the sodium alginate solution and stirred for 20 mins. Then this entire solution is been added to chitosan chloride solution by a syringe dropwise to form gel beads. The gel beads are been kept in such condition for 20 mins, after that separated via vacuum filtration and washed with distilled water. The encapsulation efficiency is been calculated by the ratio of the total phenolic content of initial phenolics dissolved into the solution for gelling minus the total phenolics content of the microgels to that of initial phenolics dissolved expressed in percentage (Moschona and Liakopoulou, 2018).

The purple sweet potato anthocyanin is been extracted and purified. 0.1 mol Cinnamic acid is been added into 20% ethanol solution at 35-40°C. Then 0.15 mol of thionyl chloride is been slowly added into the solution with stirring and distilled to remove residual salts. Then this whole mixture is been added dropwise into maltodextrin solution and stirred vigorously for 8 hrs. The resultant is been obtained as an esterified form of maltodextrin with cinnamic acid grafted like gels which are washed with distilled water and vacuum dried for further use. The antioxidant analysis of the maltodextrin grafted cinnamic acid is been done by the DPPH method. The purple sweet potato aqueous extract is been added into the wall matrix solution in a 1:2 (w/v) ratio and this entire mixture is been homogenized at 10000 rpm for 10 mins, then spray dried into microcapsules which are stored at 4°C inside falcons. The encapsulation yield is been calculated from the ratio of the final weight of microcapsules to the initial weight of the aqueous mixture. The encapsulation efficiency is been calculated by the ratio of total anthocyanin release from capsules minus superficial release of said to the total release of the said expressed in percentage. The total monomeric anthocyanins are been measured using a UV-vis spectrophotometer with help of two buffers namely 0.4 M sodium acetate and 0.025 M potassium chloride at 700 and 520 nm respectively. These anthocyanins are been expressed as cyanidin-3-glucoside per 100 g of sample (Yi et al, 2016).

Alginate and pectin are been dissolved into water in the ratios of 60:40, 70:30, 80:20, and 100:0 in (%w/v) respectively. 1mg/ml concentration of folic acid is been dispersed into an alginate-pectin solution with a stirrer. Then this mixture is been pumped via an encapsulation nozzle into gently agitated calcium chloride solution at 25°C to form folic acid microcapsules due to calcium chloride solution contact. These capsules are been then hardened for about 180 minutes by leaving them in the dark and then rinsed with Milli-Q water to wash off residual folic acid and finally stored in vials for further analysis. These capsules are then been air-dried for 24 hrs at 25°C, again freeze-dried for 6 hrs, and stored at 4°C for 11 weeks in sealed vials (Madziva et al, 2005).

III. RESULTS AND DISCUSSIONS

Self-assembly of tannic acid with FSGH has been an efficient free radical scavenger and chelator of oxidants and metal ions respectively due to enhanced absorption. Cocoa procyanidins nanoparticles have been potent apoptotic agents against leukemia cells. It can remove hazardous ions without altering the digestive activities of the gastrointestinal tract. The polyelectrolyte complex of curcumin and pomegranate ellagitannin gelatin nanoparticles has been proved to be cytotoxic toward cancer cells. The partially purified ellagitannins gelatin capsules have been less toxic and have fewer side effects compared to free ellagitannins and exhibited better retention capabilities. Intestinal epithelial cells have been serving better absorption of coated nanogels. Gelatin-lecithin capsulation on thymol has been proved to enhance bioavailability. Nanogel freeze-dried globules have higher antimicrobial stability rather than the ordinary extracts of phytonutrients. Layer by layer coating of the plant phytochemicals has been proved to be more protective for them against natural degradation. Gelatin-coated curcumin nanoparticles and alginate-gelatin polyelectrolyte resulted in sustained release of the bioactives. Gelatin-coated films have been proved to enhance the shelf life of highly degradable plant extracts in food industry applications (Ali et al, 2019).

Spray drying at 180°C has been providing the most retention of polyphenols and anthocyanins from CFE and CWE. A higher amount of release is been found in the case of maltodextrin encapsulation. A major amount of polyphenols such as anthocyanins have been achieved from the waste extract. The high encapsulation yield of polyphenols and anthocyanins has been about 63.5% and 97.8%(w/v). Maltodextrin (EE%) has been within 73.8% to 97.3% compared to skimmed milk within 27.3% to 79.1%. After digestion testing the total anthocyanin content has been reduced by 49-73% and total polyphenolic content has been reduced by 6-42% (Cujic, 2019).

Ascorbic acid content has been 134.65 ± 7.78 to 194.70 ± 7.40 mg/100g of blackberry pulp solids. The higher arrowroot starch and gum arabic concentrations result in higher encapsulation efficiency as a thermoprotective agent against drying heat for ascorbic acid content. The retention of this acid in blackberry powder has been from 68.70 % to 91.82 %. The inlet

temperature of 143°C and encapsulation ratio of 1.78:1 (arrowroot starch/gum arabic: blackberry pulp solid) have been favorable for 92% ascorbic acid retention. Anthocyanin content has been almost 79.02 ± 1.42 to 120.37 ± 2.05 mg/100g of blackberry pulp solids. Higher retentions of the anthocyanin have been found in the blackberry pulp solids at inlet temperatures of 143°C, 150°C, and 125°C with lower encapsulating agent concentration. The residence time of the powder at higher temperatures has been shorter hence the thermal degradation and oxidation of anthocyanin have been lower. Higher antioxidant activity has been produced by a higher inlet air temperature of 143°C with encapsulating agent concentration of 1.78:1(arrowroot starch/gum arabic: blackberry pulp solids) which is about 1257.34 ± 268.86 (µmol ferrous sulfate/g of blackberry solids) using FRAP assay and 161.77 ± 1.10 (g of blackberry solids/g DPPH) using DPPH assay (Noguira et al, 2020).

The recovery of ascorbic acid in the case of rice starch and gum arabic coating has been about 97-99% and 98.8% respectively. Gum arabic has been more protective than rice starch for ascorbic acid (Trinidade and Grosso, 2020).

The release of ascorbic acid from the three different coating agents used polyisobutylene, ethyl cellulose, and carnauba wax has been about 50-70%. The higher the molecular weight of the coating agent the lesser the release of acid. The initial release has been also measured and compared to that of the final one after coating, and there has been no considerable difference between the two. There has been no significant difference between those three coating agents in terms of release. In the unencapsulated form loss of ascorbic acid has been more but in the coated form it has been negligible. The spray drying temperatures have not been affecting the release of coated ascorbic acid particles. In the case of polyisobutylene coating, the release rate has been much slower and in the case of ethyl cellulose, it has been much higher. Again in the case of those of carnauba wax coating, the said have been much slower compared to the former two (Uddin et al, 2001).

UPLC chromatogram shows that maltodextrin concentration has not been affecting the polyphenol separation through the column. The retention of rosmarinic acid, eupatorin, and sinensetin compared to the initial solution of dried sample and final solution of spray-dried powder has been resulting from (65-82)%. The sample without encapsulation has been showing huge degradation of polyphenols. The retentions of rosmarinic acid, eupatorin, and sinensetin without encapsulation are 45%, 60%, and 78% respectively. UPLC analysis has been showing that the 0.05% encapsulating agent whey protein isolate concentration has been providing maximum retention of 82%, 81%, and 79% of rosmarinic acid, sinensetin, and eupatorin respectively. On contrary with 10.67% of the whey protein concentration, the retention has been reduced to 78%, 66%, and 74% due to higher protein concentration. The inside polyphenols have not been accessible due to higher protein concentration for which the retention has been decreased. The increase in maltodextrin concentration from 0.53% to 5.33% increases polyphenol retention by about 3.7%. 5.33% of maltodextrin concentration has been providing the best outcome of 82%, 80%, and 82% of rosmarinic acid, eupatorin, and sinensetin respectively. Hence it has been clarified that maltodextrin proves to be a better coating agent than whey protein (Pang et al, 2014).

The polyphenol content of the initial cocoa liquor nanoemulsion has been about 7.93 mg of epicatechin equivalent and 29.59 mg of gallic acid equivalent per gram of defatted dried mass. The radical scavenging activity of the original emulsion has been representing a median inhibitory concentration of 5.48 mg of DPPH radicals. The polyphenolic contents having antioxidant activity have been about 0.79-0.95 mg of epicatechin equivalent and 2.95-3.94 mg of gallic acid equivalent per gram of defatted dried mass respectively. The catechin and epicatechin content has been 0.25-0.29 per gram of defatted dried mass and 0.32-0.33 per gram of defatted dried mass respectively. The polyphenolic yield has been 78-93% from microcapsules. Higher retention of bioactive compounds has been found at 180°C. The antioxidant activity has been representing a median inhibitory concentration of 6.43 mg at inlet 120°C, 6.39 at inlet 150°C, and 7.18 at inlet 180°C of DPPH radicals respectively. The said has been 8.41 mg at inlet 120°C, 7.77 mg at inlet 150°C, and 5.49 mg at inlet 180°C of ABTS cationic radicals. The highest antioxidant activity has been found at 180°C from microcapsules. The storage of microcapsules has been resulting in better and higher preservation of antioxidants and polyphenols at 4°C, 25°C, and 60°C with 30 days of intermittence rather than unencapsulated cocoa liquor emulsion (Calva et al, 2019).

The entrapment efficiency of riboflavin has been quite high about 73-81% in the internal gelation method and 55-68% in the external gelation method. The 20% lipid-loaded cold-set gels have significantly higher entrapment. About 40% release of riboflavin has been found after 6 hrs of intestinal pancreatin exposure. The more the whey protein concentration has been, the more has been the retainment, whereas the more the digestion of the matrix has been, the more has been the release. Simply via diffusion within the gut, about 95% of release has been without lipid-loaded microgels. 13% release has been in the case of 20% lipid-loaded microgels. Now in the case of slow gastric digestion about 87% release has been with 20% lipid-loaded gels. Again in the case of intestinal digestion about 83% release has been from 10% lipid-loaded gels (Egan et al, 2013).

The encapsulation yield for coating concentration of 1:3.5 (ascorbic acid: alginate solution) shows the maximum yield of 60.65% at an inlet temperature of 110°C whereas the said at 2.5:3.5 (ascorbic acid: alginate solution) shows a lower yield of 32.12% at an inlet temperature of 110°C. Total ascorbic acid content has been about 92% on the initial day and 94% after 30 days and surface ascorbic acid loss on the initial day has been 6% and after 30 days is 7%, hence the encapsulation efficiency has been 93.48% at initial day and 92.55% after 30 days for the coating concentration of 2.5:3.5 (ascorbic acid: alginate solution) (Marcela, 2016).

The total polyphenolic content estimated by HPLC and ESI analysis has been about 18-22 mg/g of sample constituting ellagic, trans-fertaric, quercetin, and kaempferol majorly. The antiradical activity has been highly estimated by the DPPH assay. The release in the phenolics ranged from 44-53% by the end of 24 hrs. Alginate-chitosan has been providing maximum retention of bioactive compounds and acts as the best non-toxic polymer for coated delivery of functional components (Moschona and Liakopoulou, 2018).

The monomeric anthocyanins are been identified via HPLC-MS. It has been seen that peonidin, ferulic acid, caffeic acid, and cumaric acid are been some largely present anthocyanins. The antioxidant activity almost has increased by 40% using maltodextrin grafted cinnamic acid for anthocyanins of purple sweet potato. The encapsulation efficiency of maltodextrin grafted cinnamic acid has been more than that of only maltodextrin whereas the encapsulation yield has been less compared to the normal maltodextrin wall material. The total monomeric anthocyanins measured by two buffer systems has been about 259.1 mg/100 g of sample. The sample kept in dark conditions has been having higher anthocyanin compared to that kept in illuminated conditions. The maltodextrin grafted cinnamic acid-coated anthocyanin of spray-dried purple sweet potato extract microcapsules has been about 44.3%, 46.3%, and 71.8% higher for samples kept at dark, normal illumination, and ultraviolet condition than those kept with ordinary maltodextrin coating respectively (Yi et al, 2016).

The freeze-dried alginate pectin folic acid microcapsules have been showing improved stability. More than the air-dried capsules, the freeze-dried ones have been retaining retain folic acid. The coating concentration of (70:30) and (60:40) alginate-pectin ratio has been best in terms of higher retention of folic acid. The release of folic acid from coating concentration of (70:30) alginate-pectin has been about 90% which takes about 100-120 minutes in the intestinal juice of pH 8.2 whereas the release of said in case of only alginate coating (100:0) concentration has been about only 25 minutes in the same pH. Folic acid loss has been higher in alginate than in alginate-pectin at pH 1.2. Thus in both pH scenarios, the loss has been higher in the case of only alginate than alginate-pectin (Madziva et al, 2005).

IV. CONCLUSION

The demand for herbal supplements has been trending in this pandemic era. The covid scenario has led to ardent arousal of plant-based diets to avoid detrimental side effects of lethal doses of synthetic drugs and steroids administered to eradicate the virus. These supplementary herbal food products are been needed to be delivered with proper viability. Spray drying has been adopted for such a purpose. To make the processing more rational, the coating polymers incorporated are been providing more fundamentality for the preservation of these bioactive functional components. They not only have been protecting the sensitive core compounds against various degradation but also have been preserving them for longer shelf life. The number of bioactives found in the initial mixture prepared with coating polymers, emulsifiers, and binding agents, and that in the final powder after spray drying is almost the same even after long-term storage. They have been embedding the functional core appropriately thus delivering them to the target sites of our body even after surviving gastrointestinal digestion.

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