CHEMISTRY

DEGRADATION

OVERVIEW

Degradation reduces a product into its components through physical and/or chemical means. The methods outlined in this section are from studies interested in the degradation of plastic. Provided are the methods for organic enzymatic degradation and photodegradation by irradiation. Both induce a chemical response from the object of interest. Substances have varied responses to different degrading agents; hence, ensuring compatibility between the agent and the substance of interest is vital in drawing a favorable result.

A. Photodegradation

M. oleifera sp.

(Bastareche, Catolico, Secular, Larroder)

Forty mL of 0.03 molar concentration (M) silver nitrate was measured. The silver nitrate was stirred at 400 rotations per minute (rpm). Then, ten mL of *M. oleifera sp.* seed extract were slowly dropped into the silver nitrate using a pipette. The same procedure was also done with the synthesis of the 10-g and 15-g *M. oleifera sp.* seed extract.

LDPE films

(Agno, Gilongos, Jalandoni, Sinco)

Before light exposure, each of the 12 LDPE films was labeled with the following: TNP-UV (treated with undoped titanium dioxide nanoparticles under UV light), TNP-VL (treated with undoped titanium dioxide nanoparticles under visible light), N-TNP-UV (treated with nitrogen-doped titanium dioxide nanoparticles under UV light), and N-TNP-VL (treated with nitrogen-doped titanium dioxide nanoparticles under visible light), for a total of four experimental set-ups with three replicates each. The LDPE films were initially placed in their respective 250 mL beakers containing 150 mL of 20mM TNP and N-TNP aqueous suspensions (Kim et al. 2003). It was ensured that the tested surface of LDPE films was exposed to the suspension at all times.

Two sets of beakers containing the samples were enclosed in a 24" x 8" x 6" wooden box. The first set, which contained three replicates of TNP or N-TNP treated set-ups, was constantly irradiated using a UV- A light (18W, <320nm primary wavelength), and the second set, also containing three replicates of TNP or N-TNP treated set-ups, with a light-emitting diode (LED) bulb (9W that is equivalent to 18W-fluorescent lamp, 400-700 nm wavelength) at a 10 cm distance from the base of the beaker (Ali et al. 2016). The photodegradation process was carried out for 336 hours. During this period, the set-ups were stored in ambient air and room temperature (Tofa et al. 2018). After irradiation, LDPE films were thoroughly rinsed with distilled water.

EVALUATION

OVERVIEW

Products, whether resulting from or used for research, undergo an evaluation to ensure stability, effectivity, and safety. This section provides the evaluating methods from various studies as it relates to products such as hydrogel, bioplastic, antibacterial soap, and biodiesel. Further, parameters for oil adsorption, antioxidant property, and a makeshift reaction chamber are included. Aside from the final product, the proprietary of some parameters also requires evaluation to ensure a controlled environment throughout the study.

A. **Bioplastic Sheets**
(Rentoy, Angot, Mabaquiao, Larroder)

The first and the fourth repetitions were successful in producing solid sheets that could be tested. The first batch was tested on its mechanical properties, particularly the density, percent elongation, and tensile strength as these were the only available tests in Central Philippine University - Packaging Engineering. The fourth batch was tested of its chemical composition using the FT-IR Spectroscopy in Philippine Science High School Western Visayas Campus. All the tests were compared with commercial cellophane.

B. **Oil Adsorption**
(Janiya, Lopez, Magtoles)

The initial weight of the samples was recorded with the use of an analytical balance. The samples were submerged in crude oil for 30 minutes. Then after the adsorption process, the samples were removed with a strainer and then weighed. The amount of oil adsorbed was calculated using the formula by Tolba et al (2011):

$$qe = \left(\frac{Wo - We}{M}\right)$$

Where:

q_e = amount of oil adsorbed per unit weight of adsorbent
w_o = initial weight of oil (g)
w_e = weight of oil sample after the adsorption process (g)
M = mass of adsorbent (g)

C. **Device**
(Gurrea, Peregrino, Regalado, Salvador)

For added information, the fluid dynamics of the bubbling CO_2 may also be calculated based on the data. Utilizing 20L of CO_2 over 14.56stranslates to 1.37L/s of CO_2 being bubbled into the solution. Using the Bernoulli equation one can also derive its velocity which is equal to 4.84m/s and its pressure which is 23.24Pa or 0.003 PSI. This means that only as much as 0.003 PSI back pressure from cars is necessary to facilitate the mineral carbonation process. This shows that 1.37L/s of gas is very doable in practical as well as experimental situations since it generates only minimal backpressure.

Assuming a 1:1 ratio (i.e. a 100percent efficiency) approximately 39.32g of CO_2 will necessitate 68.87g of $Ca(OH)_2$. $Ca(OH)_2$ was set as the excess reactant and spread evenly amongst the 3 chambers and 24g of CO_2 was added per chamber for a total of 72g. Furthermore, based on previous studies, an optimal concentration of 30 ppm or 0.09g of Nickel nanoparticles were also added into the solution, evenly spread amongst the 3 chambers.

As a result of the reaction of $Ca(OH)_2$ and CO_2, $CaCO_3$ was expected to precipitate at the bottom of the chambers. The contents of each chamber were then drained, filtered and washed, oven-dried, and weighed. The precipitates were washed with distilled water while being filtered to ensure the purity of the $CaCO_3$. This was then oven-dried at 60°C overnight. These were done to determine the amount of $CaCO_3$ precipitate present in the solution.

D. Physiochemical Properties

(Hembra, Henderin, Pareñas, Sinco)

Leaves of Mangifera indica (mango) contain phytochemicals that promote antibacterial activity. This study aimed to determine whether *M. indica* leaves extract can be an alternative antibacterial agent for triclosan.

The formulated soap was subjected to multiple tests for its physicochemical properties as described below.

Measuring the pH of soap is significant in determining whether the formulated solution is corrosive to the skin or not (Vivian et al. 2014). The pH test was done in triplicates and was based on the standard protocol recommended by American Oil Chemists' Society (AOCS) (1997). Using buffer solutions of pH 4.00, 7.00, and 10.00, the EUTECH pH700 pH meter was calibrated. After calibration, the probe was dipped into 10% solutions of each of the formulated soaps. The obtained pH value was then recorded.

The density of liquid soap is influenced by the amount and molecular weight of its components (Handrayani et al. 2015). The density of each of the soap solutions was determined by first preparing 10mL of each soap solution and measuring each of the masses of the prepared solutions using the Sartorius top loading balance. The obtained mass was divided to 10mL to calculate the density.

Foam stability is dependent on the properties of the surfactant (Osei-Bonsu et al. 2015). For the assessment of foam stability, 10mL of each soap solution was measured and then mixed in a vortex for five (5) minutes. The foam height produced was measured using a ruler with +0.5mm precision. After allowing the solution to stand for 5 minutes, the foam height was again measured. Foam Stability was measured using the formula (Handrayani et al. 2015):

$$\text{Foam Stability} = \frac{\text{Final Foam Height}}{\text{Initial Foam Height}} \times 100$$

Equation 3. *The equation was used to calculate the foam height.*

Similar to pH, free caustic alkali is also responsible for the abrasiveness of the soap and is used to determine if the product is hypoallergenic or not. For the determination of free caustic alkali, 5g of each soap solution was measured and then dissolved in 30mL ethanol. After dissolution, three (3) drops of phenolphthalein indicator and 10mL of 20% barium chloride (BaCl2) were added into each of the solutions. After thorough mixing, each solution was then titrated with 0.05M H2SO4. Free caustic alkali was determined using the formula by Vivian et al. (2014):

$$FCA = \frac{0.31}{W} \times VA$$

Where W is the weight of the soap, and VA is the volume of H_2SO_4.

E. Biodiesel Properties

(Almarza, Gatila, Inosanto)

Sixteen biodiesel properties were calculated using the FAME composition of each species. Fuel properties derived from FAME profiles are the following: degree of unsaturation (DU), long-chain saturation factor (LCSF), cold filter plugging point (CFPP), iodine value (IV), saponification value (SV), cetane numbers 1 and 2 (CN1 and CN2), saturated fatty acids (SFAs), mono-unsaturated fatty acids (MUFA), polyunsaturated fatty acids (PUFA), kinematic viscosity, density, higher heating value (HHV), amount of C18:3, number of double bonds (Db), and oxidation stability.

The FAME profile of the seaweed species involved is the direct result of the analysis conducted by the GC-MS equipment. These data are used to calculate and predict the values of the parameters using their respective formula under the biodiesel standards EN 14214 and ASTM D6751-02, which will determine the biodiesel fuel quality of the organisms.

FABRICATION

OVERVIEW

Fabrication involves the design and manufacturing of a product or object. The methods in this section provide the proposed process of production and specifications for sugarcane bagasse-based mesh and bioplastic sheets. Written here are experimental methods aimed at the production of the aforementioned products. Fabrication is a rigorous process requiring multiple trials - time is an essential factor in identifying points for improvement.

A. Mesh
(Bayer, Farinas, Navarra, Yturralde)

The mesh was created by using sterile gauze as the base. The base was used because of the bagasse fibers' short length. Then, the epoxy was beforehand diluted with 100 percent denatured alcohol to effectively spread the epoxy between the fibers. The solution was then added to the mesh to hold the fibers. The mesh is then left to dry overnight. The bagasse meshes were treated with concentrated sulfuric acid for 30 minutes and then were kept in an oven at 150C for 24 hrs to activate the adsorbent.

B. Bioplastic Sheets
(Rentoy, Angot, Mabaquiao, Larroder)

The cellophane casting stage is when the viscose is poured onto the glass slides and bathed into the chemical baths of 40% ammonium sulfate and 12% sulfuric acid-18% sodium sulfate. In a 250-mL beaker, 200 g of ammonium sulfate was weighed to create 500 mL of 40% solution of ammonium sulfate. Subsequently, the sulfuric acid-sodium sulfate bath was made by adding 60 mL of sulfuric acid into a one-liter graduated cylinder. After sulfuric acid, 90 g of sodium sulfate was added. The solution produced was a 12% sulfuric acid-18% sodium sulfate solution. The two baths, ammonium sulfate, and sulfuric acid-sodium sulfate baths were heated over a hot plate up to 45◦C.

The sheet of bioplastic was prepared by spreading a thin layer of viscose on the 1x3x8 inch glass plate using a rubber spatula. The viscose was allowed to coagulate by immersing the sheets in an ammonium sulfate bath (45◦C) for 60 seconds. After the immersion, the coagulated sheet that was still on the plate was immersed in a sulfuric acid-sodium sulfate bath (45◦C) for two minutes.

Upon contact, the yellow sheets gradually turned white and a bubbling effect was observed. The cellulose-based bioplastic films were then soaked in hot distilled water (80◦C) for 10 minutes. To obtain a more flexible bioplastic, the bioplastic sheets were treated with a 5% glycerin solution for 15 minutes. The bioplastic sheets were flipped during the glycerin treatment to allow even plasticizing. The sheets were air-dried for three days at room temperature.

In the first repetition, the viscose was spread onto the inside of the glass slides, which surrounds the viscose with walls. Sheets were successfully made. In the fourth repetition, the chemical used for the first bath was ferrous ammonium sulfate instead of ammonium sulfate. Moreover, the underside of the glass plates which had no walls surrounding was used instead of casting the sheets inside the glass plates with walls. The sheets were then immersed into pans filled with coagulating and regenerating reagents. Sheets were successfully made. In repetitions three and five, the sheets would tear at every attempt to remove them from the glass since they adhered to the surface of the glass plate. The films were not appropriate for testing since they would tear easily and the sizes of the film were too small.

METHODS OF ANALYSIS

OVERVIEW

Chemical analysis is the process of quantifying and/or identifying a chemical species. This section provides broad-scope methods in analyzing Chromium (VI) and phosphates. Each species may require different methods to be accurately measured. There are various considerations in this type of analysis such as the oxidation state of the target species, phase of the substance, and the target data, e.g. quantity, concentration, etc.

A. Chromium (VI)

(Faciolan, Leonora, Majaducon, Sinco)

Chromium in its hexavalent state, Cr^{6+}, is one of the prevalent heavy metals in aquatic ecosystems with its occurrence primarily attributed to industrial activities such as dye manufacturing and construction run-off. This paper presents the removal efficiency of organo-mineral composites from the shells of three mollusks abundant in the Philippines: *Crassostrea iredalei* (Slipper Cupped Oyster), *Perna viridis* (Green Shell), and *Telescopium telescopium* (Horned Snail).

A fresh 10ppm Cr^{6+} solution was prepared for treatment. Prior to the start of treatment, the initial absorbance and pH of the stock solution were measured using Shimadzu UV-vis Spectrophotometry and a pH meter, respectively. The pH of the solution was acidified with 1M HCl to a pH level of at most 3. This was to prevent the reduction of Cr^{6+} ions to Cr^{3+} which occurs upon the addition of bases—which in this case are the mollusks' shells themselves (Sanchez-Hacchair and Hofmaan 2018). Blanks were prepared using distilled water with the same mass of shells as the treatments.

Three set-ups—composed of triplicates for each shell powder treatment, and a blank— were used for the duration of the treatment phase. Mollusk shell samples were introduced into the Cr^{6+} stock solution by adding 1.0 mg of the crushed powder to 100 mL of the solution per replicate (Abeynaike et al. 2011; Baijnath et al. 2014).

The same amount of shell powder was added to 100 mL of each blank. Scilogex SK-L180-Pro laboratory shaker was used to agitate the mixtures at 100rpm for 20 minutes (Weerasooriyagedra and Kumar 2018; Zhou et al. 2019), after which, the final absorbance and pH of each replicate were measured.

The calibration curve was used to convert absorbance data to concentration, expressed in parts per million (ppm) Cr^{6+}. The concentration of Cr^{6+} removed was obtained by subtracting the final

concentrations of the stock solution and the blank replicate to the initial concentration of the 10ppm Cr^{6+} stock solution.

Adsorption efficiency (Q) for the three species was calculated using Equation 4:

$$Q = \frac{C_0 - C_1}{C_0} \times 100$$

Equation 4. Formula for adsorption efficiency (Q)

where C_0 is the initial concentration of Cr^{6+} and C_1 is the resulting concentration following treatment.

B. Ascorbic Acid Method (For Phosphates)

(Derramas, Gonzales, Villaflor, Mediodia)

Fifteen milliliters (15 mL) of 36 N concentrated sulfuric acid (H_2SO_4) was diluted to 108 mL to form 5 N sulfuric acid. A solution of potassium antimonyl tartrate ($K_2Sb_2(C_4H_2O_6)_2$) was made through dissolving 1.3716 g of the compound in 400 mL of distilled water in a beaker. Twenty

grams (20 g) of ammonium molybdate ($(NH_4)_2MoO_4$) was also dissolved in 500 mL of distilled water. A solution of ascorbic acid was prepared through mixing 1.76 g of ascorbic acid to 100 mL of distilled water in another beaker. A combined reagent was made through mixing 50 mL of 5 N sulfuric acid, 5 mL of potassium antimonyl tartrate solution, 15 mL of the ammonium molybdate solution, and 30 mL of the ascorbic acid solution for a total of 100 mL of combined reagent that was used for the chemical analysis of the samples (Clesceri et al. 1992).

The ascorbic acid method was utilized to dye the samples a blue color proportional to the phosphorus concentration, making the samples analyzable via UV-visual spectrophotometry, a colorimetric method of analysis. Potassium antimonyl tartrate and ammonium molybdate, under the acidic conditions set by sulfuric acid, creates a chemical complex, which when reduced by the addition of ascorbic acid, dyes the samples. For each sample, 50 mL of the solution was pipetted into an Erlenmeyer flask. One drop of phenolphthalein indicator was added. If a red color developed, 5 N H_2SO_4 was added drop by drop to discharge the color. Eight milliliters (8 mL) of the combined reagent was added to the samples and mixed thoroughly. This process was repeated for each replicate for all treatments.

C. Phosphate Adsorption of Biochar (Stannous Chloride Method)

(Diaz, Golo, Villaluna, Presno-Aban)

Research regarding biochar remediation efficiency of excess nutrients such as phosphates is limited due to its low adsorption capacity. The study aimed to determine the potential of pineapple peel-derived biochar in adsorbing phosphates. Pineapple peel biochar was produced via pyrolysis at 300 °C, 400 °C, and 500 °C, and then characterized using a Fourier Transform Infrared Radiation (FTIR) spectrometer.

then oven-dried at 60° C for 12 hours. After cooling down to room temperature in a desiccator, the solids passed through a 100-mesh sieve to obtain the final biochar sample. The resulting biochar produced from each temperature were referred to as PP300, PP400, and PP500.

Three concentrations (5 ppm, 15 ppm, 25 ppm) of monopotassium dihydrogen phosphate (KH_2PO_4) solutions were prepared by dissolving respective amounts (5 mg, 15 mg, 25mg) of KH_2PO_4 in 1 liter distilled water. All glassware used was washed using 1:1 HCl and rinsed with distilled water to prevent contamination of other chemicals (APHA 1992).

In each 250 mL Erlenmeyer flask, 150 mL of phosphate solutions were placed with a total of 45 flasks with 15 flasks for each phosphate concentration (5 ppm, 15 ppm, 25 ppm). Out of the 15 flasks, 5 flasks were each added with 0.3 g of biochar pyrolyzed at the same temperature (300° C, 400° C, 500° C), and each flask was agitated for 24 hours at 200 rpm using a mechanical shaker (Yao et al. 2011). This process was repeated for all three types of biochar obtained from different pyrolysis temperatures, and for all three concentrations.

The agitated phosphate solutions were first filtered with Whattman 150 mm filter paper. Afterward, 0.45 µm nylon membrane filters which were soaked in 2 liters of distilled water for 24 hours, were used to further filtrate the remaining contaminants (APHA 1992).

The phosphate content of the treated solutions was determined using the stannous chloride acid method (APHA 1992). In a volumetric flask, 25 g of ammonium molybdate($(NH_4)_6Mo_7O_{24}$-$4H_2O$) was dissolved in 175 mL distilled water. In another volumetric flask, 280 mL of concentrated H_2SO_4 was added to 400 mL distilled water. After the solution was cooled to room temperature, the H2SO4 was added to the ammonium molybdate solution and diluted to 1 L and was then labeled “Ammonium Molybdate Solution”.

In a 100 mL beaker, 2.5 g fresh stannous chloride ($SnCl_2$ - $2H_2$) was mixed with 100 mL glycerol. It was heated using a hot plate and stirred using a glass rod to hasten dissolution. The reagent is stable and requires neither preservatives nor special storage. It was labeled "$SnCl_2$ Solution".

OPTIMIZATION

OVERVIEW

Optimization is the process of maximizing or minimizing a study parameter to make the best or most effective use out of a particular resource. This section includes methods that involve separating mixtures using a separatory funnel and a rotary evaporator. These may be used by young researchers aiming to optimize the methanolic mixtures of aloe gel and aloe latex with rind.

A. **Aloe Gel**
(Alcalde, Tajo, Valencia)

The aloe gel was mixed with 100 mL hexane and 100 mL methanol and placed in the separatory funnel. The mixture was shaken to hasten the distribution of solutes. The layered mixture was separated using the separatory funnel. The methanolic extract was placed in a rotary evaporator. A more concentrated aloe gel and the methanolic mixture were then retrieved from the rotary evaporator.

B. **Aloe Latex with Rind**
(Alcalde, Tajo, Valencia)

The aloe latex powder was dissolved in 100 mL hexane. The solution was mixed with 100 mL methanol and placed in the separatory funnel. The mixture was shaken. The mixture of two immiscible solutions was separated using a separatory funnel. The methanolic solution containing the aloe latex and rind was placed in a rotary evaporator. A more concentrated aloe latex, rind, and methanolic mixture were then retrieved from the rotary evaporator.

PRODUCTION

OVERVIEW

Production is the process of making something from raw materials. This section provides a method for the production of materials required for the implementation of the study. Specifically, this method may be used in producing carbon dioxide to trigger a reaction that would be necessary to evaluate a new device.

A. Carbon Dioxide

(Gurrea, Peregrino, Regalado)

To evaluate the device, CO_2 was necessary for the reaction to take place and hence the purchasing of a CO_2 tank. A makeshift regulator was made by using adapters, tees, hose, and hose clamps. An -in brass adapter for the CO_2 tank was fitted. This was then connected to an -in stainless steel tee. The other two junctions of the tee were connected to a pressure gauge and a customized hose nozzle. The customized hose nozzle was made by welding a to -in adapter and -in to -in hose nozzle. Thus, the resulting customized hose nozzle had -in socket and -in hose. A -in hose was then connected in the hose nozzle and was fastened by the hose clamp. Teflon was also wrapped between the adapters to ensure that no gas would leak out.

SPECTROPHOTOMETRY

OVERVIEW

Spectrophotometry is an experimental technique used in several quantitative analyses in various fields. It measures the amount of chemicals in a solution or the amount of absorbed light based on the principle of compounds absorbing or transmitting light over a particular range of wavelengths. This section provides spectrophotometric methods using various instruments namely Ultraviolet-Visible (UV-Vis) spectrophotometer, Fourier-Transform Infrared (FTIR) Spectroscopy, Microwave Plasma - Atomic Emission, and Has Chromatography - Mass Spectrometry for various studies.

A. UV-Visible

Absorbance of Chromium(VI) Solutions

(Faciolan, Leonora, Majaducon, Sinco)

Chromium in its hexavalent state, Cr^{6+}, is one of the prevalent heavy metals in aquatic ecosystems with its occurrence primarily attributed to industrial activities such as dye manufacturing and construction run-off. This paper presents the removal efficiency of organo-mineral composites from the shells of three mollusks abundant in the Philippines: Crassostrea iredalei (Slipper Cupped Oyster), Perna viridis (Green Shell), and Telescopium telescopium (Horned Snail).

The stock solution was subsequently diluted to the 100 ppm standard solution from which 1, 5, 10, and 50 ppm solutions were prepared from. This is observing the validity range for the linearity of the Beer-Lambert Law (Sanchez-Hacchair and Hofmaan 2018). The spectrum absorptions of the standard solutions were measured via the Shimadzu UV-visible Spectrophotometer from 600 to 200 nm, as prescribed by Onchoke and Sasu (2016). A line of best fit relating the absorption to concentration in parts per million (ppm) was then modeled in Microsoft Excel.

Phosphate Analysis

(Diaz, Golo, Villaluna, Presno-Aban)

Research regarding biochar remediation efficiency of excess nutrients such as phosphates is limited due to its low adsorption capacity. The study aimed to determine the potential of pineapple peel derived biochar in adsorbing phosphates. Pineapple peel biochar was produced via pyrolysis at 300 °C, 400 °C, and 500 °C, and then characterized using a Fourier Transform Infrared Radiation (FTIR) spectrometer.

The procedure for ash and biochar separation was acquired from the study of Wang et al. (2016). The resulting products from pyrolysis were immersed in 0.1 M HCl solution for 12 hours, washed with distilled water repeatedly, then oven-dried at 60° C for 12 hours. After cooling down to room temperature in a desiccator, the solids passed through a 100-mesh sieve to obtain the final biochar sample. The resulting biochar produced from each temperature were referred to as PP300, PP400, and PP500.

The phosphate analysis was conducted by batches of five to maximize the capacity of the Shimadzu Ultraviolet–visible (UV-Vis) spectrophotometer. A volume of 25 mL of the phosphate solution samples were placed in 50-mL Erlenmeyer flasks. One (1) mL of the ammonium molybdate solution was added and mixed. Then, two drops of the stannous chloride solution was added and swirled. The flask was manually shaken several times to mix the solution. After waiting between 5 to 15 minutes, samples were transferred to cuvettes, and absorbance of samples were measured using the UV-Vis spectrophotometer at a wavelength of 650 nm (APHA 1992).

A calibration curve (y = 0.0658571x+ 0.174952) was prepared using six phosphate solutions of known concentrations ranging from a blank sample to 25 ppm, with an increment of 5 ppm. The absorbance value for each concentration was measured at a wavelength of 650 nm. The values were then recorded and plotted. An absorbance versus concentration graph was constructed by making the absorbance values as the y-axis and the concentrations as the x-axis. The absorbance of the standard concentrations were plotted on the graph. A "best fit" line was drawn through the points using the free online application Desmos Graphing Calculator™. The concentration of the phosphate solution samples were interpreted using the graph.

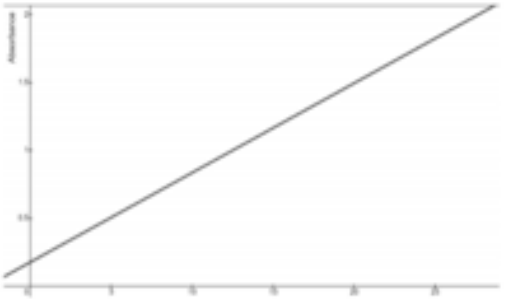

Figure 16. *Concentration versus absorbance calibration curve.*

Absorbance of *Chlorella sorokiniana* culture samples

(Derramas, Gonzalez, Villaflor, Mediodia)

After 20 minutes of exposure of the treatment solutions to the ascorbic acid method reagents, the absorbance of each sample was measured at 880 nm, using distilled water as a blank solution for reference. For every round of analysis, the cuvette was first rinsed with distilled water and then with the analyte solution. After this, it was filled to mark with the analyte solution. After properly inserting the cuvette to be analyzed into the UV-visual spectrophotometer, the absorbance of the sample was measured. This was repeated for every replicate. After the individual analysis of the samples per replicate, the absorbance of the Conway medium content added to the samples was analyzed and then subtracted from the sample absorbances, providing the values for the phosphorus remaining in the samples, disregarding the phosphorus content of that of the Conway medium.

$A_{actual} = A_{sample} - A_{Conway}$

Where: A_{sample} *= absorbance of sample*

A_{Conway} *= absorbance of Conway medium*

A_{actual} *= absorbance of sample disregarding Conway*

Characterization of Copper-Chitosan Complexes

(Loquias, Placido, Mediodia)

The synthesized copper-chitosan complexes were characterized in terms of Ultraviolet-Visible Spectra (Surface Plasmon Resonance) using a UV-1800 Shimadzu UV Spectrophotometer (Ultraviolet-Visible Spectrophotometry).

Determination of AgNP Absorbance/Yield

(Socrates, Tang, Tionko, Bautista, Padernal)

The reaction mixture samples were studied at a wavelength of 300 to 500 nm using a Shimadzu UV-1800 Ultraviolet-visible spectrophotometer. The absorbance at λ_{max} (~400-420 nm) for each sample was obtained from the spectra. The yield or concentration of NPs in the NP solution is proportional to the absorbance at λ_{max} following Beer-Lambert's Law (Paramelle et al. 2014):

$$Yield\ (in\ M) = A/L\varepsilon \quad (1)$$

where A is the absorbance at λ_{max}, L is the path length = 1 cm and ε is the extinction coefficient expressed in $M^{-1}\,cm^{-1}$.

This yield can also be expressed in number of particles per unit volume by multiplying the yield (in M) to the Avogadro's number (N_A):

$$Yield\ (in\ particles/L) = Yield\ (in\ M)\ x\ N_A \quad (2)$$

B. Fourier-transform infrared spectroscopy (FTIR)

Characterization of Pineapple Peel Biochar
(Diaz, Golo, Villaluna, Presno-Aban)

Research regarding biochar remediation efficiency of excess nutrients such as phosphates is limited due to its low adsorption capacity. The study aimed to determine the potential of pineapple peel derived biochar in adsorbing phosphates. Pineapple peel biochar was produced via pyrolysis at 300 °C, 400 °C, and 500 °C, and then characterized using a Fourier Transform Infrared Radiation (FTIR) spectrometer.

The procedure for ash and biochar separation was acquired from the study of Wang et al. (2016). The resulting products from pyrolysis were immersed in 0.1 M HCl solution for 12 hours, washed with distilled water repeatedly, then oven-dried at 60° C for 12 hours. After cooling down to room temperature in a desiccator, the solids passed through a 100-mesh sieve to obtain the final biochar sample. The resulting biochar produced from each temperature were referred to as PP300, PP400, and PP500.

The Attenuated Total Reflection (ATR) method for solids was used in order to identify functional groups using the Fourier Transform Infrared (FTIR) spectroscopy. Two grams of each biochar sample with different pyrolysis temperatures (300, 400, 500) were prepared for the analysis through powderizing the biochar using mortar and pestle. The pre-installed computer program for the test was set to identify functional groups within the 4000 to 500 cm−1 spectrum, but other preset parameters for the analysis were not changed. After the prior preparations, the samples were placed under the ZnSe crystal. After the samples were fixed for the analysis, the program was run to collect 256 scans to obtain the FTIR graph from the sample using transmittance (%). The results for each sorbent were saved and were named according to their pyrolysis level (PP300, PP400, PP500) (Wang et al. 2016).

Characterization of Copper-Chitosan Complexes
(Loquias, Placido, Mediodia)

The synthesized copper-chitosan complexes were characterized in terms of Absorbance Spectra using an IRAffinity-1S Shimadzu Fourier Transform Infrared (FTIR) Spectrophotometer.

Characterization of LDPE Films
(Agno, Gilongos, Jalandoni, Sinco)

Before the start of the exposure under ultraviolet and visible light, LDPE films were characterized using Fourier Transform Infrared (FTIR) Spectroscopy. After irradiation to ultraviolet and visible light, the LDPE films were subjected again to FTIR spectroscopy (Shimadzu IRAffinity-1). A wave range of 4000-400 cm^{-1} was used. The vibration peaks were recorded and analyzed for the chemical transformation of the films and to determine the functional groups present in the film (Ashraf 2014). The degree of chemical changes in the LDPE films in terms of its carbonyl and vinyl indices was then determined.

Microplastics Analysis
(Colacion, San Diego, Secondes, Oberio)

Petri dishes containing the obtained microplastics of size ≤2 mm from each sampling location were sent to Advanced Device and Materials Testing Laboratory (ADMATEL) in Taguig City, Metro Manila for analysis. The Perkin Elmer FTIR Spectrometer Frontier ATR FTIR model was used to determine the chemical composition of the identified microplastics. One representative microplastic piece from each sample was selected and subjected to the analysis. The microplastic particles to be analyzed were selected based on whether they can be manually handled for the procedure. The instrument was set to reflection mode with a 4000-600 cm-1 range with 20 scans at 8 cm-1 resolution. A spectra library linked to the ATR-FTIR was used to determine the identity of the acquired sample spectra.

C. Gas Chromatography- Mass Spectrometry

Analysis of Fatty Acid Composition
(Almarza, Gatila, Inosanto)

The samples for lipid analysis were stored in an icebox and transported to the laboratory of the College of Fisheries and Ocean Sciences department of University of the Philippines - Visayas (UPV) in Miag-ao, Iloilo where it was subjected to pretreatment for Chromatographic analysis. The lipids underwent derivation using 14% BF3 as reagent in order to produce Fatty Acid Methyl Esters, afterwards these lipids were to be injected in the Gas Chromatography with Mass Spectrometry (GC-MS). The process was done with the aid of the laboratory technician of UPV.

The pretreated samples were injected in the Gas Chromatography with Mass Spectrometry (GC-MS) equipment for analysis of FAMEs. The complete procedure was done using GC: Clarus 600 Gas Chromatograph; MS: Clarus 600 T-Mass Spectrometer of UPV.

Calamansi Peel Essential Oil
(Carigaba, Leonida, Masculino, Mediodia, Garbo)

Twenty (20) mL of the calamansi peel essential oil was submitted to the Department of Science and Technology - Industrial Technology Development Institute (DOST-ITDI) Standards and Testing Division Organic Chemistry Section and was subjected to Gas Chromatography Test for Limonene in order to determine the limonene content present in the product.

SYNTHESIS

OVERVIEW

To synthesize is to produce a compound by letting simpler materials react with each other. This section focuses on the synthesis of various compounds. Specifically, the following methods will help young researchers to form nanoparticles, calcium alginate-based beads, hydrogels, chitosan-hydroxyapatite composites, copper-chitosan complexes, and liquid antibacterial soaps.

A. Nanoparticles

Nickel

(Gurrea, Peregrino, Regalado, Salvador)

The chemical reduction method will be applied in the synthesis of Nickel Nanoparticles. First, 7.132 g of nickel chloride hexahydrate ($NiCl_2 \cdot 6H2O$) was dissolved in 30 mL distilled water in a 250mL beaker. After which, 16 g of sodium citrate dihydrate ($C_6H_5NaO_7 \cdot 2H_2O$) was added to the solution to act as a capping agent. The prepared solution was then placed in a water bath where it was heated (at 40°C) and magnetically stirred (at 400 rpm) for one hour. During the one-hour stirring time, a separate solution of 2.27 g sodium borohydride ($NaBH_4$) was prepared to act as a reducing agent.

This concentration corresponds to a 2:1 molar ratio of $NaBH_4$ to $NiCl_2 \cdot 6H_2O$ (Nayak et al.). The $NaBH_4$ solution was kept for temporary storage in a sealed 50 mL volumetric flask at room temperature while waiting for the $C_6H_5NaO_7$ and $NiCl_2 \cdot 6H_2O$. After an hour of stirring, the $NaBH_2$ solution was then added dropwise to the $NiCl_2 \cdot 6H_2O$ and sodium citrate solution for 10 minutes while continuously stirring at 400 rpm (at 180°C). The solution turned black, which indicated the reduction of the nickel ions. After the addition of $NaBH_4$, the temperature of the hot plate was decreased to 80°C and left constant for two hours to allow the reaction to complete.

The resulting solution was filtered using filter paper and alternately washed thrice with distilled water and ethanol. To prevent excess moisture that would contaminate the nickel nanoparticles, it was dried using a hot air oven at 80°C for one hour.

The weight of the produced nickel nanoparticles was recorded. Characterization techniques were not done for the synthesis of the NiNPs since this method has already been confirmed to work via transmission electron microscopy from the researchers' previous studies.

Silver

(Dogeno, Gamboa, Pefianco, Aban, Larroder)

40 mL of 0.03 molar concentration (M) silver nitrate was measured. The silver nitrate was stirred at 400 rotations per minute (rpm). Then, ten mL of *M. oleifera* sp. seed extract was slowly dropped into the silver nitrate using a pipette. The same procedure was also done with the synthesis of the 10-g and 15-g *M. oleifera* sp. seed extract.

Silver

(Socrates, Tang, Tiongko, Bautista, Padernal)

An 80-mL solution of 6.0×10^{-4} M ascorbic acid and 3.0×10^{-3} M trisodium citrate was stirred for five minutes at 100°C. Then, 15 mL of the silver nitrate ($AgNO_3$) solution of desired concentration was poured into the previous solution. The resulting solution was kept heated and stirred at 900 rpm using a magnetic stirrer. All experiments utilized the same process; however, variables were varied according to the values presented in Table 4.

The pH, time, and concentration values were converted to their coded factors (-1, 0, +1). Coding reduces the range of each factor to a common scale, -1 to +1, regardless of its relative magnitude. Coded factors could then be used for the design description and analyses in Response Surface Methodology (RSM) (Farahi et al. 2012).

Table 4. *Coded factors and the corresponding values for each variable.*

Variables	-1	0	1
pH	6	7	8
Digestion Time (m)	2.5	3.5	4.5
$AgNO_3$ concentration	0.005	0.010	0.015

Nanoparticle Suspension and LDPE films
(Agno, Gilongos, Jalandoni, Sinco)

TiO2 nanoparticles (TNPs and N-TNPs) aqueous suspensions at 20mM concentration were prepared by mixing 1.599g of TNPs and 1.878g of N-TNPs each with 1 L of distilled water in separate beakers. The mixtures were then ultrasonicated for 30 minutes. A total of 12 pieces of LDPE films were cut into 4 cm by 26 cm strips prior to the exposure to light (Tofa et al. 2018). Only the 4 cm by 4 cm at the center of the LDPE films were subjected to analysis leaving the remaining area touchable.

B. Hydrogel
(Gerona, Remaneses, Sorongon)

This copolymerization method was taken from the Microwave Initiated Synthesis and Application of Polyacrylic Acid Grafted Carboxymethyl Cellulose of Mishra et al. Five (5) grams of CMC was dissolved in 100 mL distilled water. Fifteen grams of Chitosan dissolved in a 100 mL solution before it was added to the CMC solution. Constituents were mixed in the reaction vessel (500 mL beaker). The reaction vessel was subsequently placed on the turntable of a microwave oven.

Microwave irradiation using American Home AMW-6510W with an operation frequency of 2450 MHz at a power of 700 W was performed for 3, 6, and 9 minutes. Periodically, the microwave irradiation was paused (as the reaction mixture started to boil, i.e. at 65 C) and was cooled by placing the reaction vessel in cold water. This was to avoid competing homopolymer formation reactions or the reaction between CMC-CMC molecules and chitosan-chitosan to the minimum and also to prevent any thermal damage to the backbone polymer chain. The reaction vessel and its contents were cooled and kept undisturbed for 12 h to complete the polymerization.

C. Chitosan-hydroxyapatite Composite
(Janiya, Lopez, Magtoles)

A ratio of 1:1 chitosan and hydroxyapatite was followed. Twelve (12) grams of chitosan was dissolved with the use of a magnetic stirrer at 600 rev/min in 250 mL distilled water with 1% v/v acetic acid. The same amount of hydroxyapatite was added slowly to the mixture while stirring. Followed by the addition of 12.5 mL of 2.5% glutaraldehyde solution to the mixture. After 90 minutes of stirring, 15 g of gelatin powder was added to the mixture while continuously stirred at 40°C. While warm, the mixture was poured into eight (8) molds which were divided into two (2) batches, batch A and B. Both batches of the composite were air-dried to form completely for 48 hours.

D. Copper-chitosan Complex
(Loquias, Placido, Mediodia)

The chitosan-copper complexes were prepared using a method adapted from Usman et al. (2012, 2013), wherein 10 mL of $CuSO_4 \cdot 5H_2O$ (0.05 M) was added to 40 mL of acetic acid solution (0.1 M) containing chitosan (0.1, 0.2, and 0.5 wt%). After constant stirring and refluxing at around 100°C-140°C for 20 minutes, a lighter blue-colored solution was obtained, and 0.5 mL of ascorbic acid (0.05 M) was added and stirred for twenty (20) minutes at room temperature.

Two (2) mL of NaOH (0.6 M) was then added, obtaining a darker blue-green solution after stirring for another twenty (20) minutes. Then, 0.5 mL of N_2H_4 (0.05M) was added, and the solution was stirred for five (5) minutes. The pH was kept at an average of 8.0 throughout the process utilizing NaOH and HCl solutions. The synthesis solution was centrifuged at 10 000 G for 10 minutes and washed with acetone (90%, v/v). The precipitate was dissolved in distilled water and vacuum dried at 50°C for 18 hours at the Regional Research Center, University of the Philippines-Visayas.

E. Liquid Antibacterial Soap

(Hembra, Henderin, Parenas, Sinco)

Leaves of *Mangifera indica* (mango) contain phytochemicals that promote antibacterial activity. This study aimed to determine whether *M. indica* leaves extract can be an alternative antibacterial agent for triclosan.

The soap formulation using hot process was based on Widyaningsih et al. (2018) with revisions. The liquid soap base was formulated by heating 21.4% (w/w) of palm oil to 80°C. The potassium hydroxide (KOH) solution was separately prepared by dissolving 4.29% (w/w) of KOH to 10.0% (w/w)of distilled water. The KOH solution was then poured into the heated palm oil. The process was exothermic so the solution was allowed to cool down to 80°C. After which, 1.07% (w/w) of sodium lauryl sulfate (SLS) and paraben were added into the solution. The soap solution was then stirred at 600rpm - 700rpm until the stir bar was no longer able to rotate. Upon reaching this state, the solution was manually stirred using a stirring rod until a semi-solid consistency was achieved. The weight of the semi-solid solution was then measured using a top-loading balance. To dilute the soap, distilled water was added to the solution while being heated.

The amount of the added distilled water was triple the recorded weight of the solution to ensure dissolution. The weight of the resulting liquid soap was measured using the Sartorius top loading balance. For the negative control, 21.4% (w/w) of the prepared soap base was set aside and stored in a properly labeled Erlenmeyer flask.

For the positive control, 4.29 x 10-5% (w/w) of Triclosan was added to 21.4% (w/w) of liquid soap base. For the preparation of the soap with the crude extract, the temperature of the soap base was first lowered to 40°C. After which, 0.536% (w/w) of M. indica extract was incorporated into the 21.4% (w/w) of liquid soap base and was mixed using a magnetic stirrer until the solution is homogeneous.