analysis using respond surface methodology was used to compute the optimal additive (CNF/GO/EO) to be added into an active film to provide the optimum mechanical properties.

Materials and Methods

Materials

Oil palm empty fruit bunch (OPEFB) fiber was retrieved from Universiti Kebangsaan Malaysia - Malaysia Palm Oil Board (UKM-MPOB) research center, Bangi, Malaysia. Sulfuric acid (Sigma Aldrich®), graphite fine powder (VChem ®), sodium nitrate (Qrec), hydrogen peroxide (GCE) were used to synthesize graphene oxide. Polylactic acid (Natureworks), chloroform (Fischer Scientific), and thyme essential oil (Sigma-Aldrich) were used as based film.

Nanomaterials preparation and characterization

Cellulose nanofiber (CNF) from oil palm empty fruit bunch (OPEFB) was prepared and characterized as described in previous method [12]. Graphene oxide nanosheet was synthesized *via* modified Hummer's method. Physical and chemical characterization of graphene nanosheet was done through field emission scanning electron microscope (FESEM) with energy dispersive X-ray spectroscopy (EDX), Fourier transform infrared spectroscopy (FTIR), and Fourier Atomic-Force Microscopy (AFM).

Polylactic acid based active nanocomposite film

In nanocomposite film making, CNF and GO were dispersed in chloroform to known concentration, the same solvent that used to dissolve PLA in this experiment. The dispersion takes place *via* solvent exchange, where the graphene oxide and cellulose nanofiber was first dispersed into acetone then into chloroform. Next, PLA pellet, CNF, GO and EO was dispersed in chloroform at temperature 60 ◦C in water bath for two hours. The mixture was cast on 19cm x 10cm acrylic plate and dried at room temperature overnight. The formed film was conditioned in desiccator for 48 hours.

Mechanical strength evaluation

Referring to method ASTM D 882-02, the mechanical strength Young modulus (Y) tensile strength (TS), and percentage elongation (%E) of prepared nanocomposite was evaluated using texture analyzer CT3 (Brookfield, USA). TA-DGA fixture accessories for packaging and thin sheet polymer was specifically used. The gauge length was set to 5 cm with crosshead speed 0.5 mm/min. In sampling preparation, films were cut into rectangular shape (8cm x 1cm). Total average of five specimens will be recorded for each test. The purpose of this test is to investigate the effect of cellulose nanofiber and graphene oxide incorporation percentage to the mechanical strength of the film that is being formed.

Experimental design

The response surface design is developed based on three factors, three level Box-Behnken. The design consists of a replicated center point and a set of points lying at the midpoint of each edge of the multidimensional cube that defines the region of interest. The processing material, independent and dependent variables involved in the design are listed in Table 1. The experimental design generated using the Design Expert Version 6.0 software. The 3-D response surface plots were also drawn using this software. The design involved 17 runs with five replications and the response variables measured were the tensile strength and elongation percentage (%E) as shown in Table 2. The nonlinear, quadratic model is given as:

$$Y = b0 + b1(CNF) + b2(GO) + b3(EO) + b12(CNF)(GO) + b13(CNF)(EO) + b23(GOEO) + b11(CNF)^2 + b22(GO)^2 + b33(EO)^2 \quad (1)$$

where y is the measured response associated with each factor level combination; b0 is an intercept; b1 to b33 are regression coefficients computed from the observed experimental values of Y; and CNF, GO and EO are the coded levels of independent variables and E is the error term. The statistical validity of the polynomials was established on the basis of ANOVA provision in the Design Expert Software. Finally, the feasibility and grid searches were performed to locate the composition of optimum formulations.