Heliyon 10 (2024) e30510

Contents lists available at ScienceDirect

Heliyon

journal homepage: www.cell.com/heliyon

Research article

The effect of maleic anhydride grafted polypropylene addition on the degradation in the mechanical properties of the PP/wood composites

Muneer Baig, Bandar Almeshari, Abdul Aabid *, Harri Junaedi, Abdulhakim Almajid

Department of Engineering Management, College of Engineering, Prince Sultan University, PO BOX 66833, Riyadh 11586, Saudi Arabia

ARTICLE INFO

Keywords:
Wood polymer composites
Polymers
Degradation
Design of experiments
Melt flow index
UV stability

ABSTRACT

This work focuses on studying the influence of coupling agents on the degradation in the mechanical properties of Polypropylene (PP)/wood composites. Maleic anhydride polypropylene (MAPP) was used as a coupling agent between the wood flour and PP matrix. As the coupling agent plays an important role in the stability of the WPC, a 10 wt% wood flour was mixed with PP granules along with a UV stabilizer and varying percentages (1, 3, 5 wt%) of MAPP in a twin-screw extruder to obtain PWC granules. The composite granules were injection molded to produce tensile samples for the mechanical characterization of the composites. To test the environmental degradation of the PWCs, the tensile samples were exposed to the environmental conditions for 0, 336 h (14 days), and 672 h (28 days) prior to testing. After the specified exposure time, the samples were mechanically characterized using tensile testing. The degradation characteristics of the WPCs were quantified in terms of the failure strains of the composite with exposure time. The experiments were designed, and various analyses, including ANOVA, regression equation, and prediction tests, were carried out to investigate the impact of parameters on the failure strain of the PWCs. Moreover, the study aimed to examine the effect of parameters such as MAPP and time, on the failure strain of the composites. From the experimental results, it is concluded that the composites containing 1 wt% of MAPP showed superior retention in the degradation of composites when compared with 3 and 5 wt% MAPP content.

1. Introduction

Since the evolution of mankind, wood has been an important natural resource and used in several ways. The prominent uses of natural wood include fuel, weapons, and as a material in construction. As an abundant and renewable material, the applications of wood have attracted scientists to study the characteristics and properties of different kinds and forms of wood for their application in the construction sector. It is reported that approximately 3.5 billion cubic meters of wood were harvested and predominantly used in casting furniture and a major portion was used in building construction [1]. On the other hand, thermoplastics are polymers that can be deformed into any shape when heated and later solidified upon cooling. The heating and cooling cycles can be repeated several times, to reshape the plastic. Since they can be molded into any desired shape, thermoplastics find their use in the fabrication of several components ranging from general consumer goods to rotary mechanical parts such as gears to components of medical equipment in

* Corresponding author.
E-mail address: aaabid@psu.edu.sa (A. Aabid).

https://doi.org/10.1016/j.heliyon.2024.e30510
Received 28 December 2023; Received in revised form 29 April 2024; Accepted 29 April 2024
Available online 3 May 2024

addition to their well-known applications in packaging and storage materials.

In the last decade, there has been a significant increase in the synthesis and applications of reinforced thermoplastic composites (TPCs). They find applications in Aerospace, Automotive, and Renewable Energy. In addition, to reinforce polymeric material with inorganic material, there has been considerable interest in the synthesis and fabrication of thermoplastic polymers incorporating wood flour and fibers as filler materials [2,3]. The wooden flour and fibers are relatively cheap and are available abundantly, thus the composites reinforced with such materials offer economically advantageous solutions [2–5]. Because of their cheap cost and ease of production, the wood flour-reinforced polymer composites find their applications in building/construction, automotive, and specifically in decking and cladding [2,4,6,7].

Wood polymer composites are currently being considered for usage in structural materials. However, the load-carrying capability of composites depends fairly on the characteristics of flour particles. The wooden flour particles tend to delaminate from the polymer matrix upon the application of the external load. The extent of debonding is dependent on the particulate size of the filler material. Usually, the flour particles are bigger and are easy to debonding from the polymer matrix thereby posing an interfacial adhesion problem [8–10]. To address this adhesion problem, some studies were performed that opted for treating wood flour particles with NaOH solution [11,12] or coating the wood flour particles with stearic acid [13,14] and other focused on using the Maleic anhydride (MA) functionalized polymers for the production of wood polymer reinforced composite [15–17]. It is reported that the stiffness of the composite is unaffected by the functionalized polymers; However, the composite showed an enhancement in its strength when using such functionalized polymers for the production of the composites [18,19]. Additionally, the tensile and the flexural strengths of the polymer composite were observed to decrease with the percentage increase of the filler material, when the polymer was not functionalized; however, these properties increased in the composite when the polymer was treated with MA or functionalized, before adding the filler material [19,20].

Additionally, some studies have been performed on functionalizing wood flour to address the adhesion problem in wood polymer composites [21]. In their study, direct fluorination of the wood flour was performed, and the fluorinated wood was later used as a filler to produce the composite. It was reported that direct fluorination led to an enhancement in the tensile and flexural properties of the developed composite. In another study, the polymer matrix (Poly Propylene) was modified with an organosilane, and the modified PP was used in producing a reinforced polymer composite. The results showed the organosilane-treated PP exhibited higher adhesion of wood flour to the PP matrix and the composite showed better mechanical properties compared to the composite where the PP was treated with MA [22]. Another study focused on treating the PP matrix with Starch Gum (SG) and later used the treated PP to produce wood/flour polymer composites. The SG-treated polymer composite was reported to perform lower than the MA-treated polymer composite in the case of the virgin PP matrix. However, the performance of SG-treated polymer composite increased in the case where recycled PP was used as a base matrix [23]. Another investigation reported the effect of pulverization on the wood flour that contained water and wood flour without water content, on its melt viscosity, and the mechanical properties of the PP/wood flour composites. The enhancement in the properties of the composite was mostly due to the particle size of the wood flour [24]. Another study focused on the effect of natural weathering on the optical properties of the transparent wood composite (TWC). The weathering on TWC was analyzed in terms of changes in visual appearance, color parameters, optical transmittance, and chemical changes. It was reported that the TWC showed degradation characteristics on weathering for 150 days. The authors used a benzotriazole-derived UV absorber (1.75 % conc.) to reduce the degradation of TWC [25]. The degradation characteristics in appearance and mechanical properties of WPC were reported in another study. The study finds that the WPC loses its properties when exposed to accelerated weathering. To avoid the degradation in properties, the authors reinforced the WPC with carbon and glass fibre-woven fabrics [26].

The method of designing experiments (DOE) has gained significant popularity in the fields of mechanical [27] and materials science engineering [28–31]. For example, DOE has been utilized by materials science researchers to determine the mechanical properties of metal matrix composites [30] and to assess the optimal composition of recycled polypropylene/rubberwood flour composites [32]. The literature discusses the implementation of the slack-variable model in a mixture of experimental designs to optimize the mechanical properties of a wood plastic composite with Polyethylene Terephthalate (PET) as the polymeric matrix. This approach involves selecting one component of the mixture as a slack variable and designing and analyzing the experiment based on the remaining components. Additionally, the compolytics-approach translates combined polymer and policy analyses into industrial design principles for thermoforming wood-plastic composites [33]. These studies demonstrate that DOE can effectively identify optimal solutions while minimizing costs and energy requirements. To utilize DOE, input variables and designs for both linear and non-linear data are required, which necessitates practical information regarding suitable parameters and factorial design. With the DOE approach, researchers can efficiently determine the optimal combination of materials and parameters to obtain desired outcomes, making it a useful tool for a wide range of engineering problems.

Based on the available literature survey, it is important to note that there have been several studies that were performed on WPCs in the last decade. However, these studies were either on the debonding of wood particles or fibers from the polymer matrix upon exposure to moisture or on the strength characteristics of the WPCs. The study on the degradation of polymers, especially the synergistic response of the polymers (inorganic) with wood (organic) on the degradation in the mechanical properties of WPCs has not been discussed in detail. Thus, in this study, the degradation of PP was minimized by selecting the optimum percentage of UVA stabilizer. Additionally, the effect of coupling agents on the degradation characteristics of the WPCs is addressed. Furthermore, the current study utilized the design of experiments to identify the optimal level of coupling agent for wood-polymer composites.

2. Materials and methods

2.1. Materials

A commercial grade (PP) Polypropylene (SABIC PP 500P) having a density of 0.905 g/cm^3, and a melt flow rate (230 °C–2.16 kg) of 3g/10 min, was used as the matrix base for the fabrication of PP/Wood composites. The PP 500P is a multipurpose-grade polymer for extrusion and injection molding applications. The filler material for the composite was wood flour (WF) extracted from the leaves of a local date palm tree. The dried leaves were initially sized and grinded to obtain a fine WF (powder). The ultraviolet light absorber (UVA) used was Tinuvin 326 supplied by BASF. It is used to improve the light stability of plastics. The coupling agent used between the PP matrix and WF was Maleic Anhydride Polypropylene (Fusabond™ P613 Functional Polymer) supplied by Dow Chemical Company.

2.2. Methods

2.2.1. Preparation of the composites

Initially, the naturally dried leaves from the date palm tree were cut and grinded to obtain a fine WF using a high-power grinder. After grinding, the WF was sieved through 300 μm mesh. This process ensures that the average size of the WF particles is less than 300 μm. The sieved WF was dried at 100 °C for 24 h in a vacuum furnace to remove any moisture content. A 10 wt% of the dried WF was mixed with varying percentages of Maleic Anhydride (Fusabond™ P613 Functional Polymer), 0.4 wt% UV stabilizer and PP granules. The mixture was subsequently extruded using a twin-screw extruder that featured three heating zones (set at temperatures of 80 °C, 200 °C, and 140 °C). The extruded filament underwent cooling when passed through an air flow device, after which it was pelletized to obtain PP/Wood composite pellets. The composite pellets were extruded again to enhance homogeneity. These composite pellets were then processed through injection molding to manufacture tensile samples intended for characterization purposes. The complete process of composite production, starting from the individual components (PP and date palm leaves), is illustrated in Fig. 1.

2.2.2. Outdoor exposure

The WPCs were exposed to outdoor environmental conditions (natural exposure) during October 2022, where the day temperature varies from 34 to 38 °C and the night temperature varies from 22 to 25 °C. The relative humidity around this time varies from 20 to 22 % with a UV index of 9. The UV index of 9 indicates very unsafe exposure to UV radiation. Additionally, the average sunshine hours around this time were 10 h.

Due to the nature of this study, the samples were subjected to maximum UV exposure for prolonged periods. The injection molded

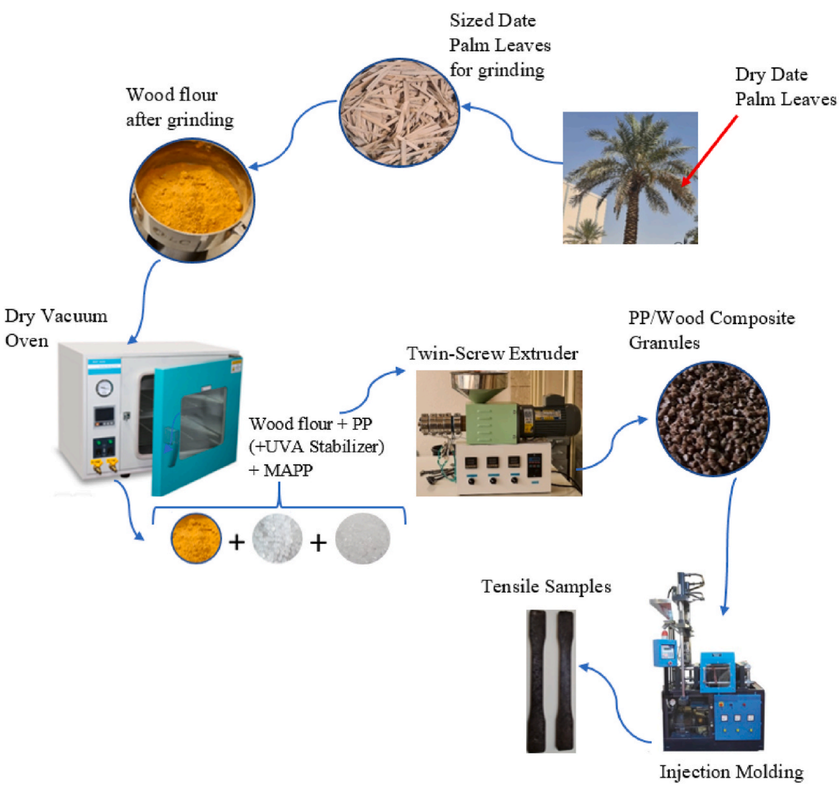

Fig. 1. Schematic representation depicting the production process of PP/Wood composite.

samples were kept in open environments for the stipulated time periods. After the stipulated exposure duration, the samples were kept in indoor environmental conditions for 24 h before testing.

2.2.3. UV spectrometry

It is well known that polymers readily absorb ultraviolet (UV) rays when exposed to sunlight. Upon absorption of UV rays, the polymers undergo degradation thereby affecting their mechanical properties. Since the WPCs are usually used in outdoor and indoor applications, it is highly desirable to protect the polymer from its degradation. In this study, Tinuvin® 326 UVA was used as a UV stabilizer to impart stability to the polymer (PP) matrix. The recommended concentration of the UV stabilizer from the manufacturer was in the range of 0.1–0.5 % for the PP matrix. Thus, the as-received PP was initially mixed with the recommended concentrations and tested for UV absorbance test using a UV spectrometer.

2.2.4. The melt flow index (MFI) of the composites

The fluidity of a polymer or polymer composite is typically assessed using a parameter called the melt flow index (MFI). This parameter quantifies the flow behavior of the polymer under a constant load and specific temperature conditions. In the present study, the MFI values of the PP/Wood composite were determined following the ASTM standard D1238. The measurements were conducted at a temperature of 230 °C and a load of 2.16 kg.

2.2.5. Mechanical characterization

The tensile strength and the failure strain values of the PP/Wood composites were determined through uniaxial tensile experiments. Fig. 2 presents the standard sample geometry employed in this study. These tensile experiments were conducted at room temperature, employing a constant crosshead speed of 5 mm/min. The load and displacement data obtained from the testing machine were utilized to calculate the tensile strength and failure strain values of the composites. To ensure statistically significant results, a minimum of three experiments were performed for each composition.

2.2.6. Optical microscopy

The optical microscope (OM) was used to observe the microstructure of the composite. The cross-section and longitudinal section of the sample were observed. For the samples to be observed under the OM, the sample surface must be flat and free of scratch. Some preparations were done for the sample. First, the sample was cut by a saw, axially and longitudinally, towards the injection molding direction of the sample. The sample cutting surfaces were then grinded with 320-grit sandpaper to remove the roughness from the cutting followed by 1200-grit sandpaper to smooth the surface. After the grinding, the sample was polished by polishing cloth to remove any scratches created by the sandpaper. The sample was then washed and dried. Finally, the polished composite sample was placed under the OM to observe its microstructure.

3. Design of experiments

In prior research, the design of experiments (DOE) method was frequently employed within the manufacturing industries [34] to determine the critical factors required for achieving goals with a satisfactory level of accuracy. These factors encompass various parameters and their corresponding levels that can be controlled by the designer. Typically, a factorial design is chosen, involving a minimum of two parameters or levels, and experiments are conducted accordingly using orthogonal arrays specifically designed for the factors under consideration. Following the orthogonal array enables the identification of each element's impact, allowing for the execution of analysis and the determination of the most effective factors for goal achievement, which can be adjusted and optimized. Therefore, in this study, the DOE method was applied to optimize the wood-polymer composite behavior of PP-wood (palm tree) based advanced composites with limited experimental work to prioritize optimization techniques, and different analyses were explored to consider further experiments.

In this study, a random orthogonal array was employed to effectively analyze the impact of process parameters on the selected response, while minimizing the number of tests required. The method utilized an orthogonal array (OA) to represent the data, with careful consideration given to factor selection and the assignment of multiple levels to each factor. It is important to ensure that the selected levels are evenly spaced. The obtained results from the experimental investigations were then analyzed using plots and ANOVA to assess the influence of each factor on the chosen response. Specifically, for this research, a total of 12 experimental runs using DOE orthogonal arrays were conducted to investigate the effect of MAPP duration on the coupling of PP-wood composites.

The random selection of tests was based on the criterion that the degrees of freedom of the orthogonal array should be equal to or

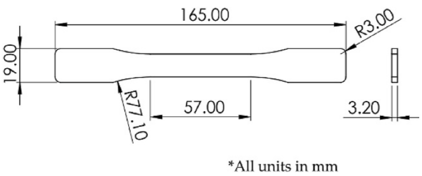

Fig. 2. ASTM (D638 Type I) standard tensile test specimen geometry was used in this study.

greater than the number of test parameters. In this experimental study, the selected parameters were determined to have a significant influence, and 12 tests were conducted, with the parameters assigned to the columns. The tests were performed using a random forest analysis. Fig. 3 depicts the methodology employed to conduct the analysis using the DOE approach, aiming to attain an optimal outcome.

To investigate the relationship between MAPP and time on the coupling behavior of the composite, a test plan was developed. The experiments were conducted by randomly combining the parameters to obtain tensile test results for various parameter combinations. The two parameters, one has 4 levels (MAPP%) and another one has 3 levels (Time Hrs.) are used in the present study, and the experimental outcomes were employed for further optimization. ANOVA, linear regression, Taguchi, and statistical analyses were performed to determine the effects of the initial interactions and achieve the optimum result.

$$Y = b_0 + b_1X_1 + b_2X_2 \tag{1}$$

The present study mainly focuses on the regression and ANOVA analyses, and the linear model is represented by Equation (1), where Y is the dependent variable (normalized strain), b_1 *and* b_2 = Linear regression coefficients of MAPP, and time. For precise results, a 12 orthogonal array is considered, which tests all levels of the other parameters with a factorial array.

Orthogonal arrays are commonly employed in industrial applications to assess the effects of different control variables. These arrays consist of columns representing independent variables, and multiple levels and factors are defined to describe them. For this study, a regular 12-row orthogonal array was chosen to conduct the tests. In Table 1, column 2 was allocated for MAPP (%), while column 3 represented time (in hours). A total of 12 tests were allocated accordingly, resulting in a cumulative degree of freedom (DF) of 11 for the experiment.

3.1. Normalized strain for DOE

In the process of optimization research, the primary focus lies on the strain as the key factor in analyzing the strength of the material model within the experimental setup. Specifically, the experimental model involves subjecting the dog bone material to dynamic uniaxial tensile stress, with consideration for relatively small dimensions. Consequently, for the purposes of the present work, an assumption is made that the material operates under plane stress conditions. To achieve the optimization of parameters based on a selected combination of material properties, the normalized strain is computed. This involves calculating the ratio between the failure strain in the wood polymer composite and the failure strain in a neat polymer (without wood). Equation (2) represents this normalized strain as follows.

$$Normalized\ strain = \frac{failure\ Strain\ in\ the\ wood\ polymer\ composite}{failure\ strain\ in\ neat\ polymer} \tag{2}$$

To enhance the data using DOE, Minitab 18 and Design Expert 13 tools were employed. The goal was to calculate the normalized strain to reduce numerical values and thereby identify the genuine material quality of the wood-polymer composition.

4. Results and discussion

4.1. UV spectrometry

Based on the recommendations from the manufacturer, the percentage of UV stabilizers in the PP matrix varied from 0.2 to 0.5 %. This was done to stabilize the degradation of the PP matrix to be used in the production of PP/wood composites. The results from the UV Spectrometer are shown in Fig. 4, where the UV absorbance is plotted vertically against the wavelength for the varying percentages of UV content in the PP matrix.

From the figure, it is observed that the UV absorbance of as-received PP is quite low. However, this absorption is sufficient for the quick disintegration of the PP polymer chains, resulting in the loss of its mechanical properties. The UV stabilizer absorbs the UV rays

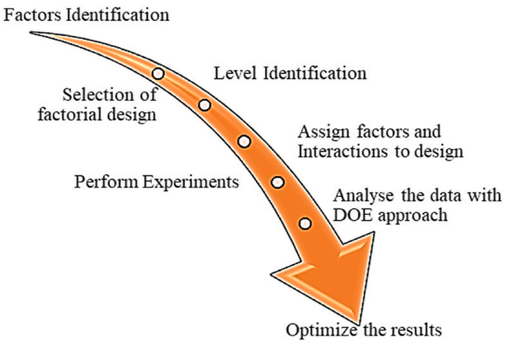

Fig. 3. Steps followed for optimization.

Table 1
Experimental runs.

S. No.	MAPP (%)	Time (Hrs.)
1	0	24
2	0	336
3	0	672
4	1	24
5	1	336
6	1	672
7	3	24
8	3	336
9	3	672
10	5	24
11	5	336
12	5	672

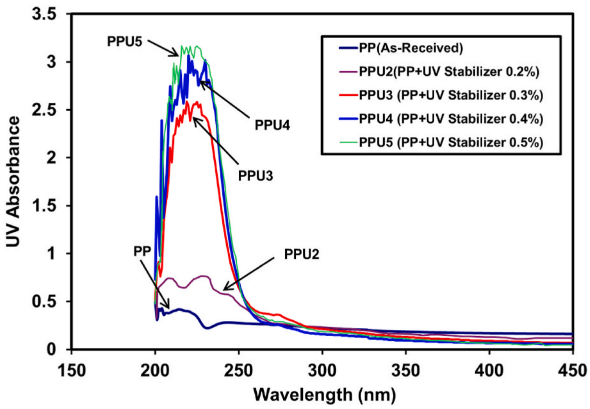

Fig. 4. The UV Absorbance responses for the varying percentages of UV stabilizer content in the PP matrix.

more rapidly than the PP matrix, and the disintegration resistance of the UV absorbers is usually much higher than the PP matrix. Thus, the presence of UV stabilizers helps in improving the life span of the PP matrix. Based on this understanding it is observed from the figure that the UV absorption of the PP increases with the increasing percentage of the stabilizers. There is a significant increase in the absorbance values by varying the stabilizer content from 0.2 % to 0.3 % and a substantial increase from 0.3 % to 0.4 %. However, the absorbance difference between the 0.4 % and 0.5 % of stabilizer content is not that significant and it also reaches the maximum concentration recommended by the manufacturer. Thus, based on these two findings, we have concluded to use a 0.4 % UV stabilizer to produce the PP/wood composites. Table 2 below shows the sample nomenclature followed for the composite code and composition used in this study.

4.2. Melt flow index (MFI)

The melt flow index (MFI) data for the PP/wood composites is shown in Fig. 5. The figure shows the variation in the MFI values with varying percentages of MAPP content in the composite. It is observed that the MFI of the composites increased as a function of MAPP content. It should be noted that the percentage of the filler material (wood flour) is constant in all the composites. Thus, one would expect the MFI values to remain constant. However, the MFI of the composite increased in this study and the change is attributed to the percentage content of MAPP in the composites. The increase in MFI of the composite implies that the MAPP assisted in the successful bonding of the wood flour particles to the PP matrix.

Table 2
Nomenclature followed in this study for the various PP/wood composites (PWC).

Code	Base Matrix	Tinuvin® 326 UV Stabilizer	Wood Flour (Filler Material)	MAPP Compatibilizer (Coupling agent)
Neat	PP	0.40 %	–	–
PWC 0	PP	0.40 %	10 wt%	0 %
PWC 1	PP	0.40 %	10 wt%	1 %
PWC 3	PP	0.40 %	10 wt%	3 %
PWC 5	PP	0.40 %	10 wt%	5 %

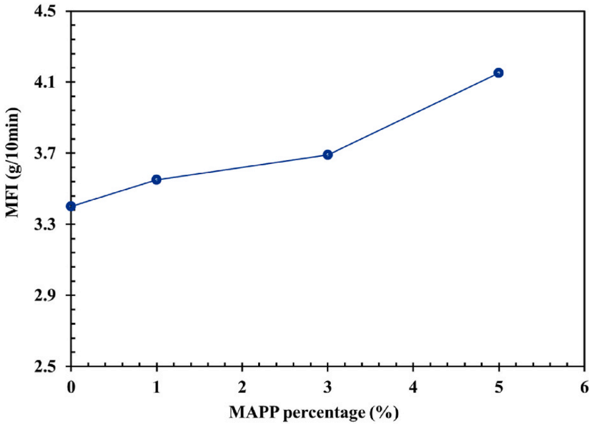

Fig. 5. Variation in Melt Flow Index (MFI) values with the percentage of MAPP content.

4.3. Microstructures of PP/wood composites (PWC)

The Optical Microscopy (OM) image of PP/wood composites along the sample cross-section containing two different compositions (1 wt % and 3 wt %) of MAPP is shown in Fig. 6 and the figure has been illustrated in two different categories 5× (Fig. 6a) and 10× (Fig. 6b) magnification for 1 wt % MAPP and similarly for 3 wt % MAPP of 5× (Fig. 6c) and 10× (Fig. 6d) magnification. From the cross-sectional view, it is observed that the wood flour particles were homogeneously dispersed in the PP matrix. At higher magnifications, the wood particles appeared to be agglomerated at certain regions marked as agglomerated wood particles (AWP) in Figs. 6 and 7.

Fig. 7 shows the longitudinal section of the composites containing 1 (Fig. 7a & b) and 3 (Fig. 7c & d) wt.% MAPP for two different magnifications. From the figure, it is evident that most of the filler material (wood) is in the form of powder (or flour). Since the wood flour was sieved using a 300 μm mesh size sieve, there were very few longer particles. Nonetheless, we have not seen any particle greater than 300 μm in length. Additionally, it is also observed that most of the wood particles aligned themselves along the loading direction or the longitudinal direction of the sample.

4.4. Tensile testing of PWC samples

Tensile experiments were performed on fabricated PWCs containing varying percentages of MAPP content. The experiments were conducted at room temperature with a crosshead speed of 5 mm/min. Before tensile testing, the PWCs were exposed to harsh environmental conditions by suspending the samples in an open atmosphere for a predetermined time. In this study, the samples were experimented with after 24, 336, and 672 h of environmental exposure. In general, most polymers when exposed to UV rays undergo photooxidative degradation resulting in the production of free radicals. The photo-oxidative degradation causes polymer embrittlement thereby resulting in loss of its mechanical properties. In this study, the degradation characteristics of the composites were quantified by strain to failure of the samples after environmental exposure. The experimental responses of the different PWCs are shown in Fig. 8. The tensile stress-strain response of Neat (PP + 0.4 wt% UV stabilizer) is shown in Fig. 8a.

From Fig. 8a, it is observed that the failure strain in the neat samples after 24 h of exposure was 800 % while the failure strain after 672 h (4 weeks) of exposure reduces to 638 %. The percentage reduction in the strain during this time interval is around 25 %. Additionally, for the responses of the PWC 0 composites shown in Fig. 8b, the percentage reduction in failure strains for the same time interval as the neat samples is found to be around 35 %. The differences in the failure strain between the responses of neat samples and PWC 0 samples are attributed to the incorporation of 10 wt% wood flour. Organic wood particles do not interact freely to form a bond between themselves and the PP matrix. Thus, it is expected that the addition of wood particles to the PP matrix would result in a loss in the percentage of failure strains of the composite. This is also inferred when comparing the results of PWC 0 with neat samples exposed to environmental degradation for 672 h, wherein the percentage change in the failure strain is around 20 %. The first two Fig. 8a and b demonstrate the importance of coupling agents in controlling the failure strains of the composite. Fig. 8c shows the stress-strain response of PWC 1 (with the addition of 1 wt% MAPP compatibilizer) composite after the exposure. Here, the percentage difference in the failure strain between the 24- and 672-h exposure samples is found to be 26 %. This value is very close to the percentage reduction in Fig. 8a, which represents the degradation of neat polymers. Thus, it can be inferred that the addition of MAPP results in good interfacial adhesion between the organic wood flour particles and the inorganic PP matrix. Similar results can be inferred from the results shown in Fig. 8d. The experimented composites in this figure were loaded with 3 wt% MAPP. The change in the percentage reduction in the failure strain between 24- and 672-h exposure is found to be around 53 %. Additionally, the percentage change in failure strains shown in Fig. 8e (with the addition of 5 wt% MAPP) for the same interval is found to be around 52 %. The results from Fig. 8d and e indicate that the percentage addition of MAPP has reached its optimum level for such composites or this level of filler material.

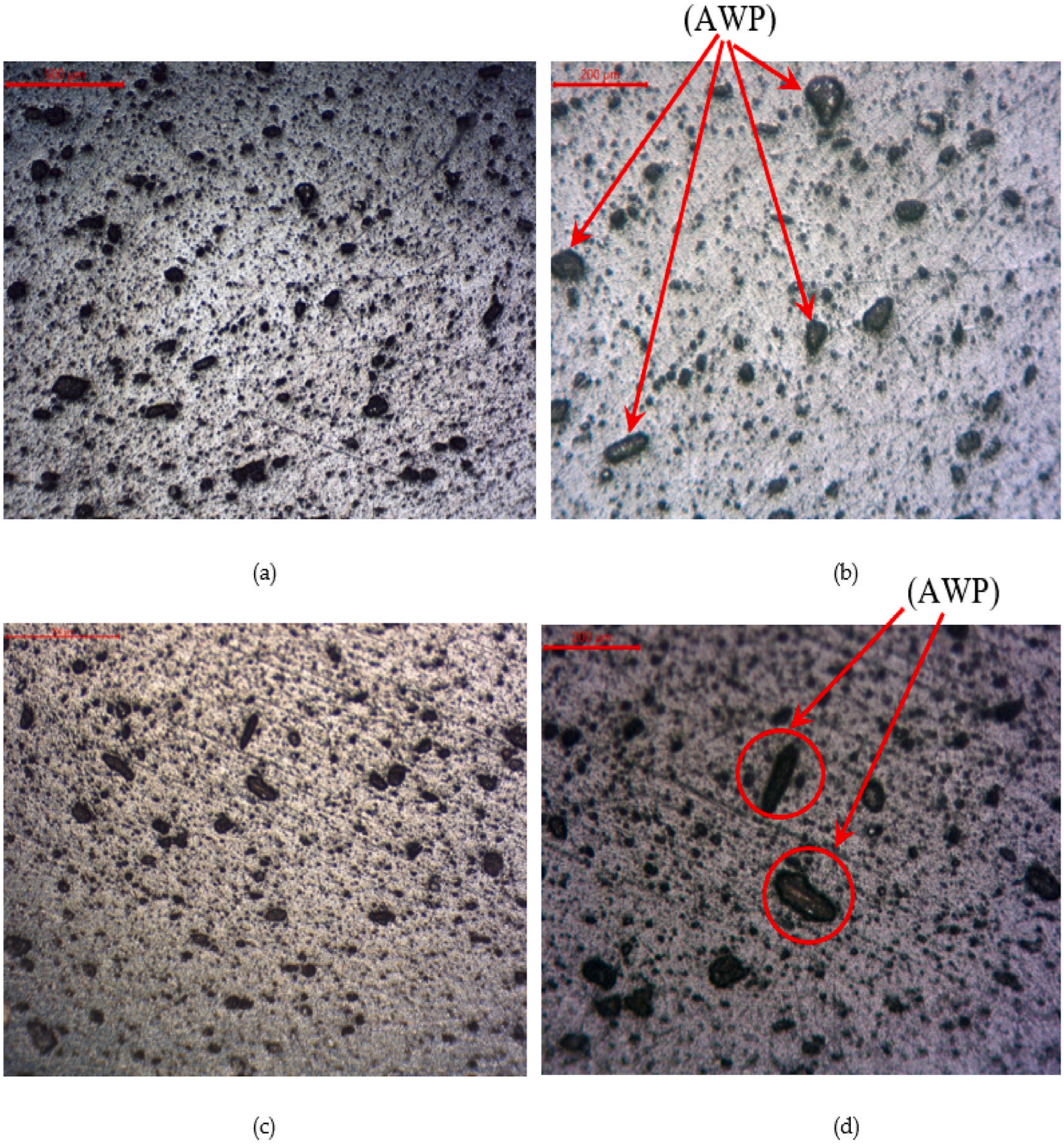

Fig. 6. OM for cross-section surfaces of (a) WPCs containing 1 wt% MAPP at a 5× magnification (marked line represents 500 μm), (b) WPCs containing 1 wt% MAPP at a 10× magnification (marked line represents 200 μm), (c) WPCs containing 3 wt% MAPP at a 5× magnification (marked line represents 500 μm), (d) WPCs containing 3 wt% MAPP at a 10× magnification (marked line represents 200 μm).

Fig. 9 shows the comprehensive bar graph of failure strains for all the composites formulated in this study. The vertical axis of the graph represents the percentage (%) of failure strain, and the horizontal axis refers to the exposure time of the composite before tensile testing. As discussed earlier in Fig. 8, the addition of a coupling agent (MAPP) is highly recommended for the long-term stability of the PWCs. Without the coupling agent, the interfacial bonding between the wood flour particles and the PP matrix is substantially low. From Fig. 9, it is observed that the addition of 1 wt% MAPP results in the failure strain in the PWC 1 close to the neat polymer. It should be noted that the failure strain achieved by the neat sample would be the maximum strain achievable because the addition of polar filler material to the non-polar polymer matrix would result in a decrease in the elongation percentage of the composite. This greatly depends on the percentages of the filler material, coupling agent, and importantly the compatibility of the coupling agent with the filler material and the polymer base matrix. Thus, based on the achieved results shown in Fig. 9, it is concluded that the PWC 1 composite has the optimum combination of material parameters to produce PWCs with long-term degradation resistance to environmental exposure.

Fig. 10 shows the variations in the yield strength of the PWCs with the MAPP content for different environment exposure times such as 24 h (Fig. 10a), 336 h (Fig. 10b), and 672 h (Fig. 10c). The change in the strength of the fabricated composites is attributed to the MAPP content. In general, the strength in the composites arises due to the interfacial adhesion between the filler material and the base matrix. The strong adhesion results in an improvement in the strength of the composite. In this study, the addition of 1 wt% MAPP and

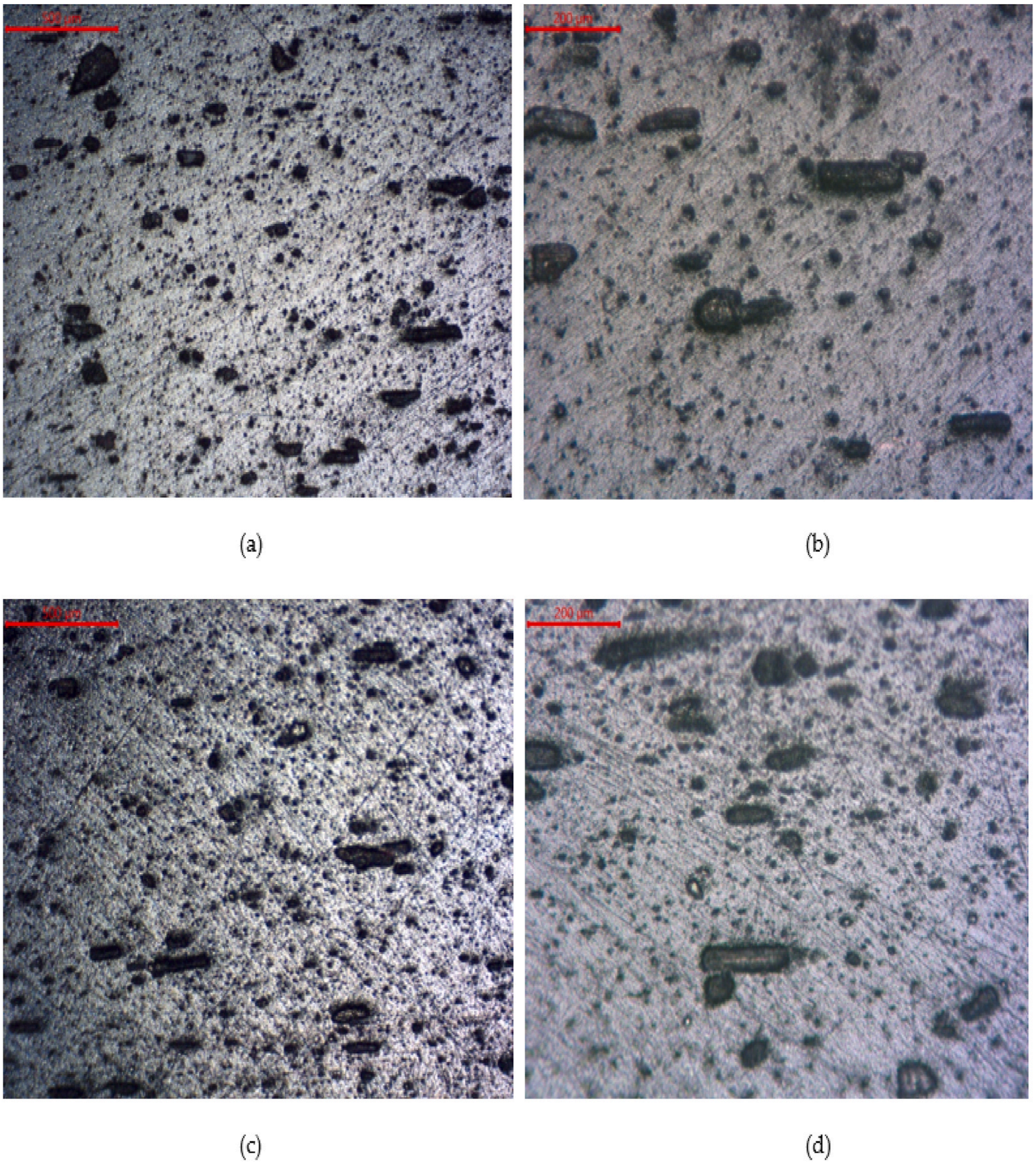

Fig. 7. OM for longitudinal section surfaces of (a) WPCs containing 1 wt% MAPP at a 5× magnification (marked line represents 500 μm), (b) WPCs containing 1 wt% MAPP at a 10× magnification (marked line represents 200 μm), (c) WPCs containing 3 wt% MAPP at a 5× magnification (marked line represents 500 μm), (d) WPCs containing 3 wt% MAPP at a 10× magnification (marked line represents 200 μm).

3 wt% MAPP results in enhancing the strength of the composite for all exposure conditions. Whereas the addition of 5 wt% MAPP results in decreasing the strength of the composite. Additionally, the increase in the strength between PWC 3 and PWC 1 is found to be relatively close to each other. However, the percentage elongation strain is significantly higher for PWC 1. Thus, based on the experimental results it can be concluded that the addition of coupling agents is mandatory in the production of PWCs. However, in this study the addition of 1 wt% MAPP results in the production of PWCs with enhanced degradation resistance.

4.5. Analysis of Variance (ANOVA)

Through ANOVA (Analysis of Variance) analysis, the effects of various factors and their relationships on normalized strain inaccuracies are assessed at specific confidence levels. ANOVA enables the identification of how each factor contributes to the overall variability of the outcomes. The outcomes of all normalized strain test cases are then presented in Table 3, employing ANOVA as the analytical approach. The analysis was conducted at a significant level of 10 percent, corresponding to a confidence level of 90 percent. The contribution of each component of the factors to the overall variance is indicated in the third column of each ANOVA table. The

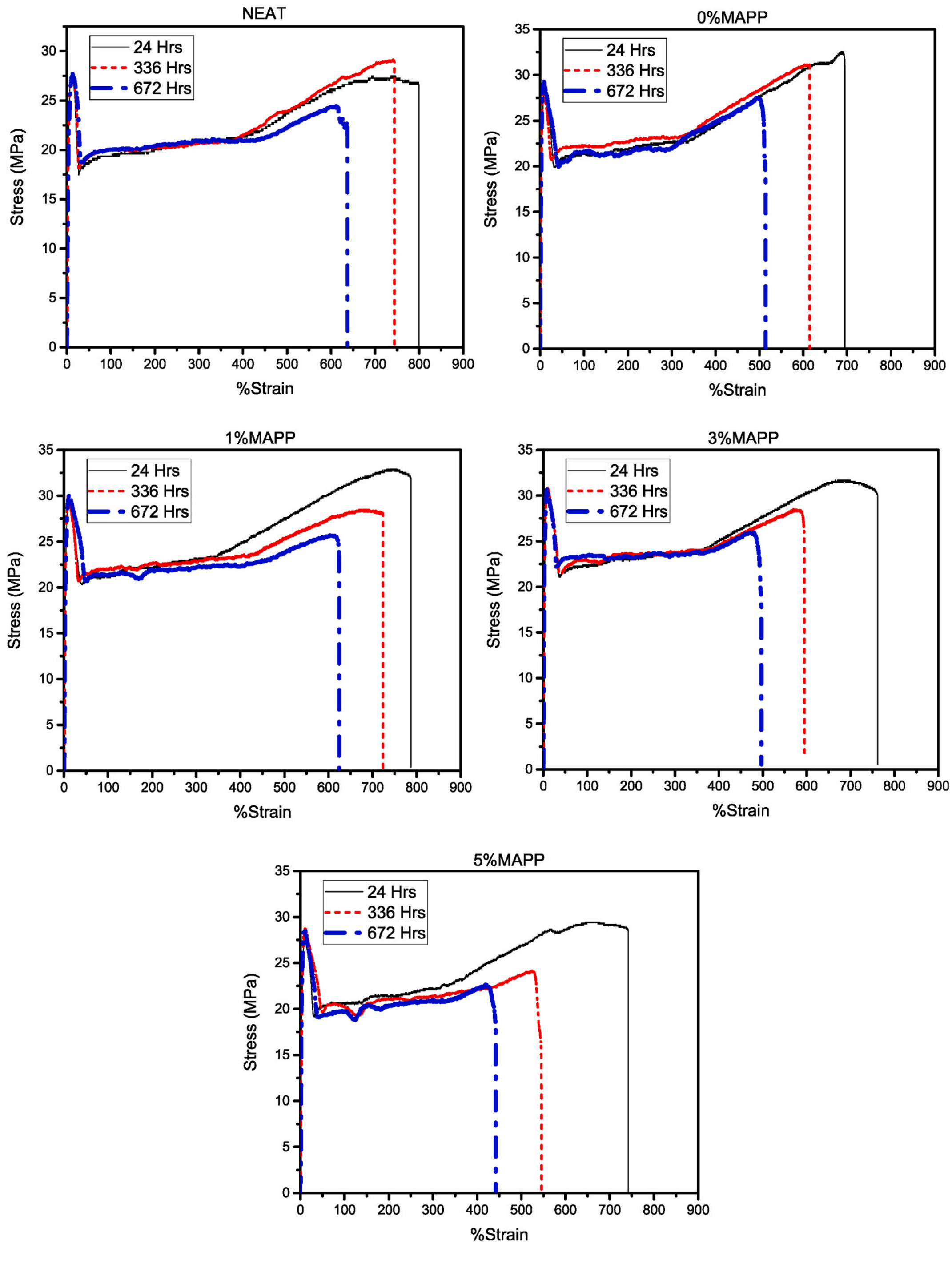

Fig. 8. Tensile stress-strain responses for (a) Neat, (b) PWC 0, (c) PWC 1, (d) PWC 3, and (e) PWC 5.

ANOVA table allows for the assessment of factors that significantly affect the response variable and factors that significantly influence normalized strain.

ANOVA is utilized to assess coupling parameters like MAPP and time impact tensile tests. The outcomes from Table 3 demonstrate normalized strain values obtained from ANOVA analysis. When the F-value exceeds 8, the corresponding parameter is deemed statistically significant. The table reveals that during the wood polymer coupling, both MAPP ($P = 0.004$) and time ($P = 0.037$) significantly affect normalized strain, with MAPP being the most influential at 71.06 %, followed by a 19.26 % influence of linear time. The ANOVA table has an associated error of 9.67 %. This method effectively showcases the variation of means and variance in absolute values considered during the experiment, confirming that coupling has a substantial impact on normalized strain values in the tensile

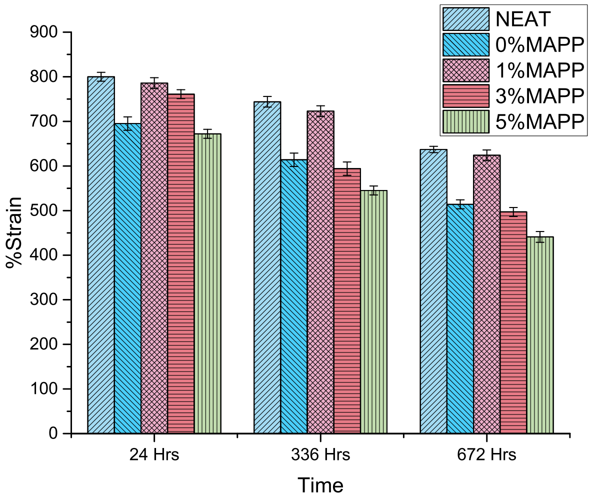

Fig. 9. Percentage strain at break of PWC samples as a function of time (hours).

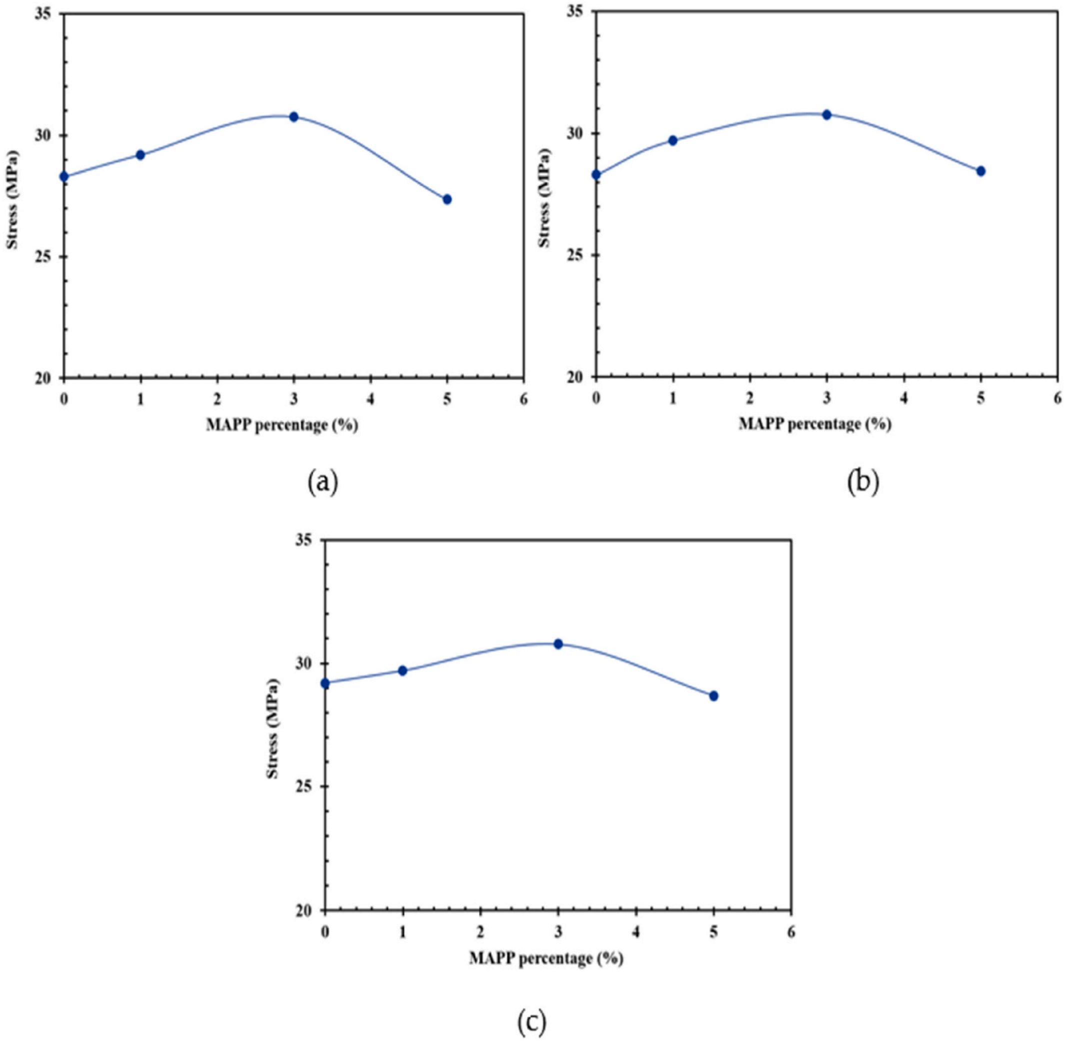

Fig. 10. Variation in the strength of PWCs as a function of MAPP content after (a) 24 Hours (b) 336 Hours and (c) 672 Hours of exposure.

Table 3
ANOVA results for mechanical normalized strain.

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
MAPP (%)	3	0.07709	71.06 %	0.07709	0.025697	14.69	0.004
Time (Hrs)	2	0.02090	19.26 %	0.02090	0.010448	5.97	0.037
Error	6	0.01049	9.67 %	0.01049	0.001749		
Total	11	0.10848	100.00 %				

SS: sum of square; MS; mean square; DF: degree of freedom.

test. Consequently, the experimental and DOE results are considered accurate.

An important component of the optimization study involves examining factor coefficients. Table 4 provides these coefficients, along with their interactions, to identify the most influential factors. The table reveals that the MAPP factor holds the greatest significance, with a coefficient value of 0.1255 at a 1 % level, followed by 24 h with a coefficient value of 0.0582. The coefficient values for the factor levels display minimal differences and a considerable distance from one another. Notably, the sign of the coefficients is not crucial; rather, their weight or magnitude is the key consideration. Please note that the t-value measures the ratio between the coefficient and its standard error. The p-value is a probability that measures the evidence against the null hypothesis and some of the other notations specified in Table 4.

The ANOVA analysis yielded a linear polynomial model, presented below, that represents the relationship between normalized strain and the variables MAPP and time. The high predicted R-Square value (90.33 %) indicates that the model is capable of accurately predicting new observations, as it fits the sample data well. The regression equation, determined using MINITAB software and the 12 experimental orthogonal arrays, is provided below. Each material combination test is represented by a linear polynomial model (regression equation) based on the corresponding components. The regression equations for these tests are as follows:

$$\begin{aligned}\text{Normalized strain} = {} & 0.8525 - 0.0188\ \text{MAPP}\ (\%)\_0 + 0.1255\ \text{MAPP}\ (\%)\_1 - 0.0092\ \text{MAPP}\ (\%)\_3 \\ & - 0.0975\ \text{MAPP}\ (\%)\_5 + 0.0582\ \text{Time (Hrs)}\_24 - 0.0205\ \text{Time (Hrs)}\_336 - 0.0377\ \text{Time (Hrs)}\_672\end{aligned} \tag{3}$$

Fig. 11 illustrates the normalized strain values obtained from the experimental results using the linear polynomial model Equation (3) for twelve test cases. The results indicate that an increase in MAPP results in a decrease in normalized strain due to the combination of organic and inorganic materials. The normalized strain performance is slightly affected by the higher level of MAPP when combined with other parameters, indicating a weak coupling between the PP and wood compared to the original material. However, this allows for the selection of MAPP under certain limits. For example, test cases 4, 6, and 7 have a maximum error compared to other test cases, but the results are higher and closer to the original material. It was also observed that the value of normalized strain decreases with an increase in the parameter level after a certain time, leading to lower repair performance of the wood-polymer composite material. As discussed earlier, the experimental results indicate that the pure composite has higher performance, but the addition of wood and polymer helps support the environment and prevents the waste of wood, which ultimately saves on material costs. The error between the predicted model fit and the experimental results is less than 6 % for all tests (as seen in Table 5). Therefore, it can be concluded that the proposed prediction model is suitable for normalized strain prediction under a certain limit of selected parameters.

4.6. Analysis of plots

The study examined how different combinations of parameters affect normalized strain. The MINITAB 18 and Design Expert software were used to generate the main product plot, which is shown in Fig. 12. This plot illustrates the impact of controlled process parameters on the normalized strain. If the line for a particular parameter is almost horizontal, it implies that the setting has little or no significant effect. Conversely, if the line for a benchmark has a steep slope, it suggests a significant impact. Based on the plot, the load has the most considerable contribution, while MAPP has a moderate impact under a certain limit. Time has a marginal effect compared to the other MAPP parameters for coupling the material's specimen. The highest normalized strain can be observed at neat 1 % at

Table 4
Coefficients list.

Term	Coefficient	SE Coefficient	T-Value	P-Value	VIF
Constant	0.8525	0.0121	70.61	0.000	
MAPP (%)					
0	−0.0188	0.0209	−0.90	0.403	1.50
1	0.1255	0.0209	6.00	0.001	1.50
3	−0.0092	0.0209	−0.44	0.676	1.50
5	−0.0975	0.0209	−4.66	0.003	*
Time (Hrs)					
24	0.0582	0.0171	3.41	0.014	1.33
336	−0.0205	0.0171	−1.20	0.276	1.33
672	−0.0377	0.0171	−2.21	0.069	*

SE: standard error; VIF: variance inflation factor.

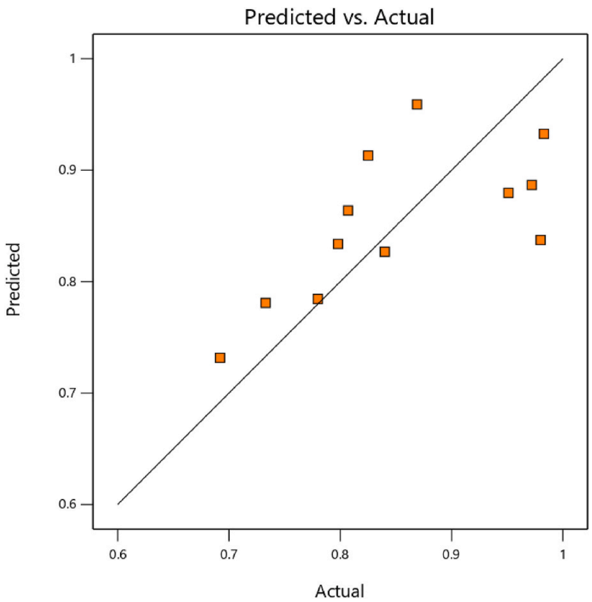

Fig. 11. Prediction plot actual vs predicted.

Table 5
Fits and diagnostics for all observations.

Obs	Normalized strain	Fit	SE Fit	Resid	Std Resid	Del Resid	HI	DFITS
1	0.8688	0.8918	0.0296	−0.0231	−0.78	−0.75	0.5	−0.75093
2	0.8253	0.8132	0.0296	0.0121	0.41	0.38	0.5	0.37867
3	0.8069	0.7959	0.0296	0.0110	0.37	0.34	0.5	0.34245
4	0.9825	1.0361	0.0296	−0.0536	−1.81	−2.46	0.5	−2.46227
5	0.9718	0.9575	0.0296	0.0143	0.48	0.45	0.5	0.44986
6	0.9796	0.9403	0.0296	0.0393	1.33	1.45	0.5	1.44615
7	0.9513	0.9015	0.0296	0.0498	1.68	2.12	0.5	2.11671
8	0.7984	0.8228	0.0296	−0.0244	−0.83	−0.80	0.5	−0.80112
9	0.7802	0.8056	0.0296	−0.0254	−0.86	−0.84	0.5	−0.83596
10	0.8400	0.8131	0.0296	0.0269	0.91	0.89	0.5	0.89386
11	0.7325	0.7345	0.0296	−0.0020	−0.07	−0.06	0.5	−0.06023
12	0.6923	0.7172	0.0296	−0.0249	−0.84	−0.82	0.5	−0.81992

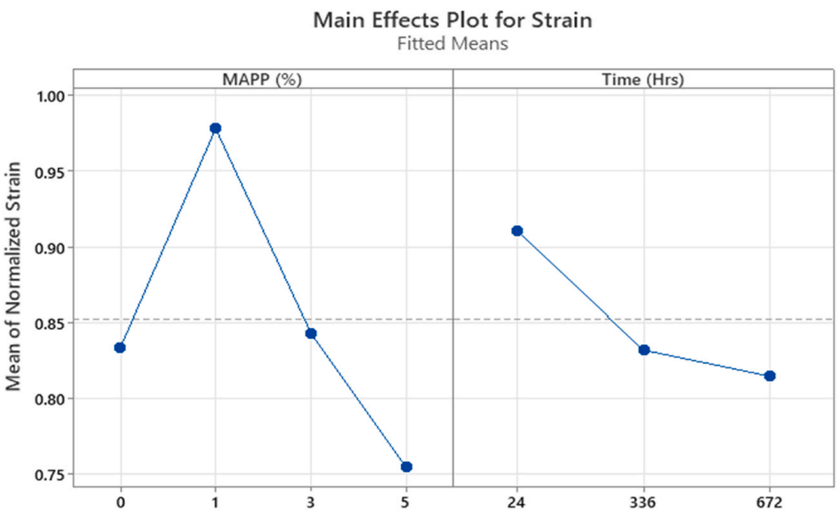

Fig. 12. Main effects plot for normalized strain.

MAPP with 24 h Of time.

Fig. 13 displays the influence of two variables on normalized strain values, indicating their increment or decrement. For each combination, the normalized strain value decreases as the level of parameters increases. It can be observed from Fig. 13 that the normalized strain losses supplement the maximum influence of MAPP with time. This is further supported by the increasing trend of reinforcement with MAPP, which does not have any significant impact on the coupling between the wood and polymer.

Interaction plots were obtained for all possible combinations of parameters to examine the difference in normalized strain and the impact of coupling between wood and polymer. Fig. 14 illustrates the slight changes in normalized strain due to the reinforcement of polymer with wood, which is consistent across experimental runs 4 to 7. Similar variations were observed in other levels of parameters, but their results showed more changes than those observed in the behavior of interaction plots. The goal of combining parameters was to achieve a normalized strain of 1. However, normalized strain drastically decreases when it goes above 1. The interaction plots reveal that changes in parameters significantly influence normalized strain, which is a similar phenomenon observed in other reinforced materials.

4.7. *Response optimization: normalized strain*

The study also included a response optimization analysis to determine the best parameter values for achieving optimal results. The primary motivation for conducting this research was to investigate the coupling agent of wood-polymer composites. As such, the objective for response optimization was to achieve maximum response normalized strain value for the MAPP. Table 6 presents the upper limit of normalized strain values predicted for all test cases. Although the target and upper limit values exhibit some variation, achieving a MAPP value greater than 1 % is not feasible for a perfect bond between the polymer and wood due to the presence of organic and inorganic materials, which makes achieving perfect bonding difficult. Based on the study, a MAPP value of 1 % results in a secure and reasonable value for normalized strain and ensures good coupling between the two agents. Therefore, the optimal parameter combination for achieving this is a MAPP value of 1 % and a time of 24 h, as shown in Table 6.

5. Conclusion

In this study, the effect of coupling agent addition on the environmental degradation of the PWC is studied. PWCs were fabricated using wood flour as filler material and PP as a base matrix. To improve the interfacial bonding between wood flour and PP matrix, MAPP is added to the mixture in varying percentages (1, 3, and 5 wt%). The PWCs were exposed to environmental degradation for 24, 336, and 672 h. The sample after pre-determined exposure is tensile tested to measure the failure strains to quantify the degradation behavior of the PWCs. Based on the experimental results it is concluded that.

1. The addition of a UV stabilizer to the as-received PP showed an increase in the UV absorbance.
2. The addition of MAPP has increased the MFI values of the composite.
3. The percentage reduction in failure strains of the PWC 0 (containing 0 % MAPP) samples exposed to 24 h and 672 h was found to be around 35 %.

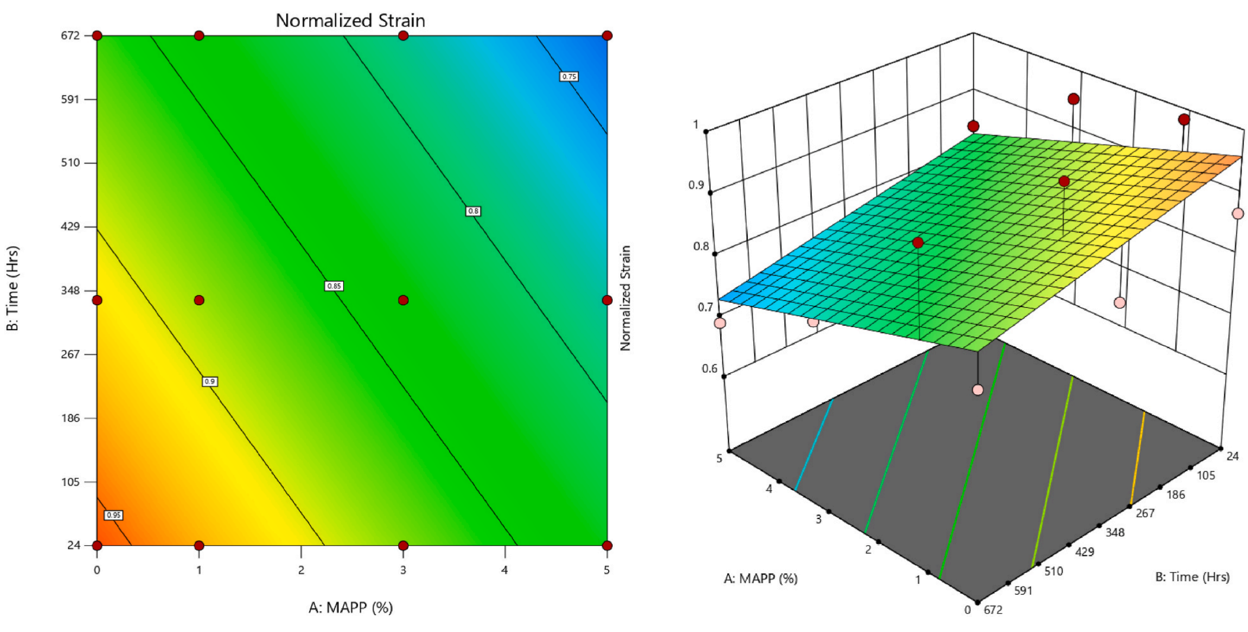

Fig. 13. Contour plot for normalized strain.

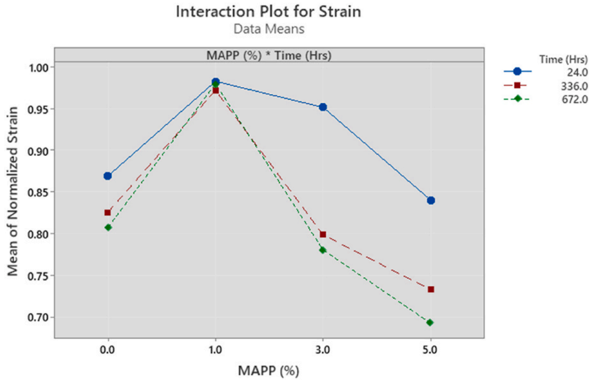

Fig. 14. Interaction plot for normalized strain.

Table 6
Response optimization: normalized strain.

Response	Goal	Lower	Target	Upper	Weight	Importance
Normalized strain	Maximum	0.692308	0.9825		1	1
Solution						

Solution	MAPP (%)	Time (Hrs)	Normalized strain Fit	Composite Desirability
1	1	24	1.03612	1

4. The percentage reduction in the failure strains of the PWC 1 (containing 1 % MAPP) samples exposed to 24 and 672 h is found to be 26 %.
5. The optimum percentage of MAPP addition to the PWC for enhanced degradation resistance was found to be 1 wt%.
6. The addition of 1 wt% MAPP and 3 wt% MAPP resulted in enhancing the strength of the composite for all the exposure times. However, the addition of 5 wt% of MAPP has resulted in a decrease in the strength of the composite for all the exposure times.
7. From the DOE, it has been revealed that the optimal parameter combination for achieving this is a MAPP value of 1 % and a time of 24 h.
8. Also, the DOE extracted that the proposed prediction model is suitable for normalized strain prediction under a certain limit of selected parameters.

Author contributions

Harri Junaedi: Visualization, Formal analysis, Conceptualization. Abdulhakim Almajid: Writing – review & editing, Supervision, Resources. Muneer Baig: Writing – original draft, Project administration, Investigation, Funding acquisition, Conceptualization. Bandar Almeshari: Investigation, Formal analysis, Data curation. Abdul Aabid: Writing – original draft, Software, Methodology, Conceptualization

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests.

Acknowledgment

The authors would like to acknowledge the support of Prince Sultan University for funding this project through seed project 77-SEED-2021-CE-77. The authors would also like to thank Prince Sultan University for paying the Article Processing Charges (APC) of this publication. Also, the authors wish to acknowledge Eng. Abdul Wasay, an undergraduate student who worked on this project for his contribution towards making the wood flour from the dried date palm leaves.

References

[1] M. Cruise, WPC Gains Ground at Domotex Hannover 2016, Floor Covering Weekly, 2016.
[2] O. Faruk, A.K. Bledzki, H. Fink, M. Sain, Biocomposites reinforced with natural fibers : 2000 – 2010, Prog. Polym. Sci. 37 (11) (2012) 1552–1596, https://doi.org/10.1016/j.progpolymsci.2012.04.003.

[3] A.K. Bledzki, M. Letman, A. Viksne, L. Rence, A comparison of compounding processes and wood type for wood fibre — PP composites, Compos. Part A Appl. Sci. Manuf. 36 (2005) 789–797, https://doi.org/10.1016/j.compositesa.2004.10.029.
[4] A.K. Bledzki, S. Reihmane, J. Gassan, Thermoplastics reinforced with wood fillers : a literature review, Polym. Plast. Technol. Eng. 37 (4) (2006) 451–468, https://doi.org/10.1080/03602559808001373.
[5] M.N. Ichazo, C. Albano, J. Gonz, Polypropylene/wood flour composites: treatments and properties, Compos. Struct. 54 (2001) 207–214.
[6] N. Ayrilmis, J.T. Benthien, H. Thoemen, Effects of formulation variables on surface properties of wood plastic composites, Compos. Part B 43 (2) (2012) 325–331, https://doi.org/10.1016/j.compositesb.2011.07.011.
[7] N. America, Cars from bio-fibres, Macromol. Mater. Eng. 291 (2006) 449–457, https://doi.org/10.1002/mame.200600113.
[8] X. Hao, X. Yi, L. Sun, D. Tu, Q. Wang, R. Ou, Mechanical properties , creep resistance , and dimensional stability of core/shell structured wood flour/ polyethylene composites with highly filled core layer, Constr. Build. Mater. 226 (2019) 879–887, https://doi.org/10.1016/j.conbuildmat.2019.07.329.
[9] E. Pérez, L. Famá, S.G. Pardo, M.J. Abad, C. Bernal, Tensile and fracture behaviour of PP/wood flour composites, Compos. Part B Eng. 43 (2012) 2795–2800, https://doi.org/10.1016/j.compositesb.2012.04.041.
[10] L. Dányádi, J. Móczó, B. Pukánszky, Effect of various surface modifications of wood flour on the properties of PP/wood composites, Compos. Part A 41 (2) (2010) 199–206, https://doi.org/10.1016/j.compositesa.2009.10.008.
[11] H. Kallakas, et al., Effect of chemical modification of wood flour on the mechanical properties of wood-plastic composites, Agron. Res. 13 (3) (2015) 639–653.
[12] M.D.H. Beg, K.L. Pickering, Fiber pretreatment and its effects on wood fiber reinforced polypropylene composites fiber pretreatment and its effects on wood fiber reinforced, Mater. Manuf. Process. 21 (2007) 303–307, https://doi.org/10.1080/10426910500464750.
[13] L. Jiang, C. He, X. Li, J. Fu, Com wear properties of wood-plastic composites pretreated with a stearic acid-palmitic acid mixture before exposure to degradative water conditions, Bioresources 13 (2) (2018) 3817–3831.
[14] S. Antiago, T.I.F.U.O. Ng, S.O.M.A.C. Hakraborty, Fabrication and characterization of high-density polyethylene – coconut coir composites with stearic acid as compatibilizer, J. Thermoplast. Compos. Mater. 23 (May 2010) (2010) 361–373, https://doi.org/10.1177/0892705709344560.
[15] N. Sombatsompop, C. Yotinwattanakumtorn, C. Thongpin, Influence of type and concentration of maleic anhydride grafted polypropylene and impact modifiers on mechanical properties of PP/wood sawdust composites, J. Appl. Polym. Sci. 97 (2005) 475–484, https://doi.org/10.1002/app.21765.
[16] R. Khamedi, M. Hajikhani, Investigation of maleic anhydride effect on wood plastic composites behavior, J. Compos. Mater. 0 (0) (2018) 1–8, https://doi.org/10.1177/0021998318816769.
[17] X.U. Min, L.I. Shuai, Impact of coupling agent on properties of wood-plastic composite, Front. For. China 2 (3) (2007) 347–349, https://doi.org/10.1007/s11461-007-0056-6.
[18] D.J. Gardner, Y. Han, L. Wang, D.J. Gardner, Wood – plastic composite technology, Curr. For. Reports 1 (2015) 139–150, https://doi.org/10.1007/s40725-015-0016-6.
[19] W. Qiu, F. Zhang, T. Endo, T. Hirotsu, Preparation and characteristics of composites of high- crystalline cellulose with polypropylene : effects of maleated polypropylene and cellulose content, J. Appl. Polym. Sci. 87 (2003) 337–345.
[20] J. Rao, Revealing the interface structure and bonding mechanism of coupling agent treated WPC, Polymers 10 (266) (2018) 1–13, https://doi.org/10.3390/polym10030266.
[21] K. Charlet, F. Saulnier, M. Dubois, A. Béakou, Improvement of wood polymer composite mechanical properties by direct fluorination, Mater. Des. J. 74 (2015) 61–66, https://doi.org/10.1016/j.matdes.2015.02.012.
[22] S.M.B.N. Ã, G.S. Cerveira, S.M.L. Rosa, polymeric-coupling agent for polypropylene/wood-flour composites, Polym. Test. 26 (2007) 619–628, https://doi.org/10.1016/j.polymertesting.2007.03.007.
[23] D.B. Rocha, S. Rosa, Coupling effect of starch coated fibers for recycled polymer/wood composites, Compos. Part B 172 (December 2018) (2019) 1–8, https://doi.org/10.1016/j.compositesb.2019.05.052.
[24] M. Haque, et al., Advanced Industrial and Engineering Polymer Research Melt-viscosity and mechanical behaviour of polypropylene (PP)/wood fl our composites : effect of pulverization of wood fl our with and without water, Adv. Ind. Eng. Polym. Res. 2 (1) (2019) 42–50, https://doi.org/10.1016/j.aiepr.2018.11.001.
[25] P. Bisht, H.C. Barshilia, K.K. Pandey, Effect of natural weathering on optical properties of transparent wood composite, Polym. Degrad. Stab. 221 (Mar. 2024) 110674, https://doi.org/10.1016/J.POLYMDEGRADSTAB.2024.110674.
[26] S. Durmaz, Y.Z. Erdil, O. Ozgenc, Accelerated weathering performance of wood-plastic composites reinforced with carbon and glass fibre-woven fabrics, Color. Technol. 138 (1) (2022) 71–81, https://doi.org/10.1111/cote.12572.
[27] A. Aabid, Y.E. Ibrahim, M. Hrairi, Optimization of structural damage repair with single and double-sided composite patches through the finite element analysis and Taguchi method, Materials 16 (4) (2023) 1581.
[28] M. Adamu, Y.E. Ibrahim, O. Elalaoui, H. Alanazi, N.M. Ali, Modeling and optimization of date palm fiber reinforced concrete modified with powdered activated carbon under elevated temperature, Sustain. Times 15 (8) (2023), https://doi.org/10.3390/su15086369.
[29] M. Adamu, Y.E. Ibrahim, M.M. Abdel daiem, H. Alanazi, O. Elalaoui, N.M. Ali, Optimization and modelling the mechanical performance of date palm fiber-reinforced concrete incorporating powdered activation carbon using response surface methodology, Materials 16 (8) (2023), https://doi.org/10.3390/ma16082977.
[30] A. Aabid, M.A. Murtuza, S.A. Khan, M. Baig, Optimization of dry sliding wear behavior of aluminium-based hybrid MMC's using experimental and DOE methods, J. Mater. Res. Technol. 16 (2022) 743–763, https://doi.org/10.1016/j.jmrt.2021.12.005.
[31] H. Alshahrani, T.A. Sebaey, M.M. AwdAllah, M.A. Abd El-baky, Multi-response optimization of crashworthy performance of perforated thin walled tubes, J. Compos. Mater. 57 (9) (Feb. 2023) 1579–1597, https://doi.org/10.1177/00219983231159508.
[32] C. Homkhiew, T. Ratanawilai, W. Thongruang, The optimal formulation of recycled polypropylene/rubberwood flour composites from experiments with mixture design, Compos. Part B Eng. 56 (2014) 350–357, https://doi.org/10.1016/j.compositesb.2013.08.041.
[33] D. Friedrich, Thermoforming of wood-plastic composites: a compolytics-approach translating combined polymer and policy analyses into industrial design principles, Int. J. Adv. Manuf. Technol. 124 (10) (2023) 3533–3551, https://doi.org/10.1007/s00170-022-10760-9.
[34] D.C. Montgomery, Design and Analysis of Experiments, eighth ed., 2012.