2025年臺灣國際科學展覽會
優勝作品專輯

作品編號 030034

參展科別 化學

作品名稱 Utilization of Nano cellulose from date palm waste for improvement of thermal stability in epoxy composite

得獎獎項 成就證書

就讀學校 First Secondary School In Safwa
指導教師 Mohamad Nurul Azman Bin Mohammad Taib
作者姓名 Zahra Hussain Ali Al Ibrahim

نموذج تفاصيل المشروع

Project Detail Form

Utilization of Nano cellulose from date palm waste for improvement of thermal stability in epoxy composite

Warning تحذير

الرجاء عدم تضمين أي معلومات شخصية عنك

Please do not include any personal information about yourself

ويشمل ذلك الصور الفوتوغرافية والأسماء

including photographs and names

القسم ١: مقدمة

SECTION 1: INTRODUCTION

(على سبيل المثال، المشكلة ومدى تأثيرها على المجتمع)

(e.g., The problem and the impact of the problem)

Nano additives is becoming popular trends nowadays due to its nanosize (1-100 nm). Incorporating nano additives in polymer could increase different properties such as mechanical, physical, electrical and thermal stability (1, 2). Different nano additives has been used such as nano copper oxide, nano silica, nano zinc oxide, nano titanium dioxide but most of these come from synthetic or metal oxides that considered as non-environmentally friendly and harmful to human when exposed or inhaled (3). One of the green materials that become attention by researchers is nano cellulose. Nano cellulose can be extracted in different methods and sources such as from wood, and non-woody resources such as kenaf, jute, bamboo as well as from bacteria such as *Acetobacter* species(4). This making nano cellulose abundantly available in resources. Nano cellulose can be in the form of nano crystalline cellulose (CNC) or NCC or can be in form of nano fibrillated cellulose (NFC) and bacterial nanocellulose (BNC)(5). This nanocellulose has many advantages that can give improvement in different applications such as mechanical, physical, thermal and improving the biodegradation when added together in different matrices (6, 7). Polymers have a problem in thermal stability while processing. It hard to control and maintain the thermal stability of polymer during processing and most polymers considered to have low in thermal stability except for thermosetting polymers such as epoxy. Epoxy has been widely used in many fields such as coating, adhesive, laminates, castings and many more (8). But the drawbacks of epoxy while using is hard to maintain and controll the thermal properties when processing of this materials and used for long period due to aging and attack by free radicals causing by UV radiation (9, 10). In this study we are incorporating nano additives into epoxy as polymer matrix to enhance and improve the thermal stability of composite by crosslinking the polymer chains with the nano additives. Furthermore, the nano additive used is come from nano cellulose extracted from date palm waste and thus to create an environmentally friendly and sustainable nano additives products.

القسم ٢: مراجعة الأدبيات

SECTION 2: Literature Review

(على سبيل المثال، مراجعة الأدبيات والخلفية العلمية)

(e.g., Literature Review and Scientific Background)

The thermal degradation of polymer is one of important aspect that need to be studied in order to determine the upper limit of temperature used in any kind of materials. This also include the mechanism of degradation into a solid state of the system (8). The attention regarding the thermal stability is directly focus on exploitation of thermogravimetric data to determine the kinetic parameters. Thus, the thermogravimetric analysis of TGA has become an important widely used technique because of its simplicity and direct information of sample afforded in thermogram. The TGA concept is based on mass loss versus function of temperature or time under controlled atmosphere (8).

The nanocellulose is one of green material that has been explored and used widely by researchers due to its abundantly available resources, high in mechanical properties, good in thermal stability and physical properties (11). The nanocellulose can be extracted in different resources such as from wood and non-woody materials as well from certain bacteria that produce cellulose. The used of strong mineral acid solution such as hydrochloric acid and sulfuric acid is the most common method in extracting nanocellulose in large scale or industrial scale (12).

The Saudi Arabia is one of the main producer of date and date products and have millions of date palm trees planted. The estimation of more than 23 million of date palm trees across the Saudi Arabia land has been planted to date (13). The trees produce a lot of wastes from pruning activity and after harvesting with the estimation around 345,000 tons wastes have been produced annually (13). The average tree can produce 15-20 kg of biomass per anum (14). The wastes come from dry leaves, frond, empty fruit bunches, trunk that always has been dumped or disposed in the landfills or burned without further utilize and this create environmental pollution (15). As in **Fig. 1** shows the parts of date palm tree that would produce the wastes. The wastes from date palm is ideal as a main raw materials or as alternative for solid wood sources in extracting nanocellulose.

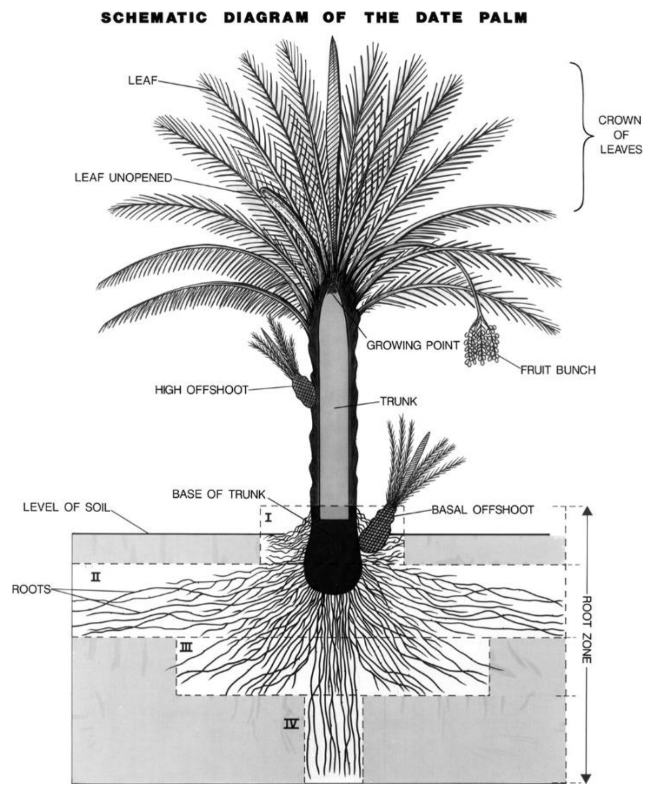


Fig. 1 Date palm tree schematic (15)

Morais et al. (16) studied on extracting nanocellulose from cotton linters with different characterization such as electron microscopic analysis, thermal analysis, X-ray diffractrometry, light scattering and contact angle. They found that the nanocellulose extracted using acid hydrolysis, increased the crystallinity index and hydrophilicity of nanocellulose. On average the nanocellulose produced from this process was around 177 nm long and 12 mm wide. More active surface means more potential to be attached in different matrix structure. The sulfate groups that come from sulfuric acid could reduce the thermal properties. Poor thermal stability will be obtained by used of sulfuric acid in hydrolysis of nanocellulose whereas the use of HCl acid with no surface charge attached to the nanocellullose structure could enhance the thermal stability of material.

In a different study by Csiszar et al. (17) that produced a suspension of nanocrystalline cellulose (NCC) from bleached cotton by controlled sulfuric acid a low frequency of ultrasound (20 kHz) and 60% different amplitude for 0, 1, 2, and 10 min. The series of film manufactured form the NCC suspensions and based on transmission electron microscopy and laser diffraction analysis proved that sonicated improved and disintegrated the larger NCC aggregated to individual nanocellulose with average of length and width of 171 ± 57 and 17 ± 4 nm, respectively. The use of ultrasound assisted disintegration to nano-sized cellulose and decreased the optical haze of suspensions with values of 98.4% to 52.8% with increasing of time from 0 to 10 min, respectively. The sonication provide a more homogenous mixture within matrix thus increased the bonding as well contributed to good tensile properties. In another study by Jamaluddin et al. (18), examined the addition of cellulose nanofiber (CNF) into poly (lactic acid). The CNF produced using mechanical process and without used of any chemicals. This significantly reduce the manufacturing cost and time processing. Then, the modification of surface nanocellulose was done with acetylation and results showed the higher hydrophobicity was obtained around 85.9° as compared with neat PLA with 70.2° contact angle. Moreover, tensile strength improved by 25% with addition of 1 wt% CNF.

القسم ٣: الأهداف

SECTION 3: Objectives

(على سبيل المثال، أسئلة البحث، والغرض من البحث والأصالة و/ أو الفرضية)

(e.g., Research Questions ,Purpose ,Novelty, and/or Hypothesis)

ضع النص الخاص بك هنا إما باللغة العربية أو الإنجليزية

The research question for this study is what is the effectiveness of nano cellulose cross-linking within epoxy chains in improving the thermal stability

The main objective and purpose for this study is to Investigate the thermal stability of nano cellulose in epoxy matrix and increase thermal stability by utilizing nano cellulose as nano additive

The specific objectives for this study are:

1. To synthesize and characterize the nanocellulose extracted from date palm waste as nano additives
2. To fabricate with different composition of nanocellulose (1%,2% and 3%) in epoxy composite

Hence, the novelty of this study is green extraction of nanocellulose from date palm waste and utilizating the green material into epoxy as a composite

The hypothesis for this study is if the nano cellulose mixed with epoxy , epoxy's thermal stability will be improved

القسم ٤: المنهجية

SECTION 4: METHODOLOGY

(على سبيل المثال، المتغيرات والضوابط والمواد والإجراءات وطريقة التقييم/النموذج الأولي)

(e.g., Variables, Controls, Materials, Procedures, and evaluation method\prototype)

Methodology

Materials

The raw materials (date palm fronds) were collected from nursery area in King Fahd University of Petroleum and Minerals (KFUPM), Dhahran. The chemicals used were sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), hydrochloric acid (HCl), epoxy and hardener (Jointmine 905-3S) were obatined from local suppliers.

Methods

Firstly, the date palm fronds were cutting into smaller size using chipper and sun dried for a week until completely dried. Then, this date palm fronds were ground into fine fibers using pulverized machine (around 50 mesh in particle size). After that, the first step of treatment was done to remove extractive from this material. The hot water with temperature around 50 °C was added three times into the fibers (5 mg) in a beaker and filtered using Buchner funnel filtration to separate the fibers and extractive solution. Then, the filtered fibers were placed in round bottom flask and added with 4% NaOH solution and treated for 2 hours to remove the lignin and other carbohydrates compounds (hemicellulose). This treatment was repeated for 3 times to make sure the removal of these components was completely done. After that, the fibers were filtered and treated with 30% H_2O_2 solution in 250 mL round bottom flask for 4 hours for bleaching process. After completed the bleaching process the fibers were filtered and cellulose fibers were obtained. The cellulose fibers were kept in refrigerator for further used and characterization.

The acid hydrolysis treatment was done with addition of around 1 mg of cellulose fibers to 5M of HCl. This treatment was done at temperature of 100 °C for 1 h to get nano cellulose. After the treatment completed, the nano cellulose was centrifuged at 7500 rpm for 20 min and rinsed with distilled water a few times until became neutral in pH. The nanocellulose was dried in oven at 50 °C for 24 hours before further used.

Fabrication of composite was done with different loading of nanocellulose 1%, 2% and 3% that was added into epoxy resin. The ratio for epoxy resin and hardener was 3:1. The nanocellulose and epoxy resin was mixed using mechanical stirer and was sonicated for 3 minutes before poured into the petri dish as former. Then, the composite was leave for 2 days for completed drying. Silicone spray was used in the petri dish as a release agent to facilitate the removal of sample. Fig. 2 shows the flow chart diagram for this experiment.

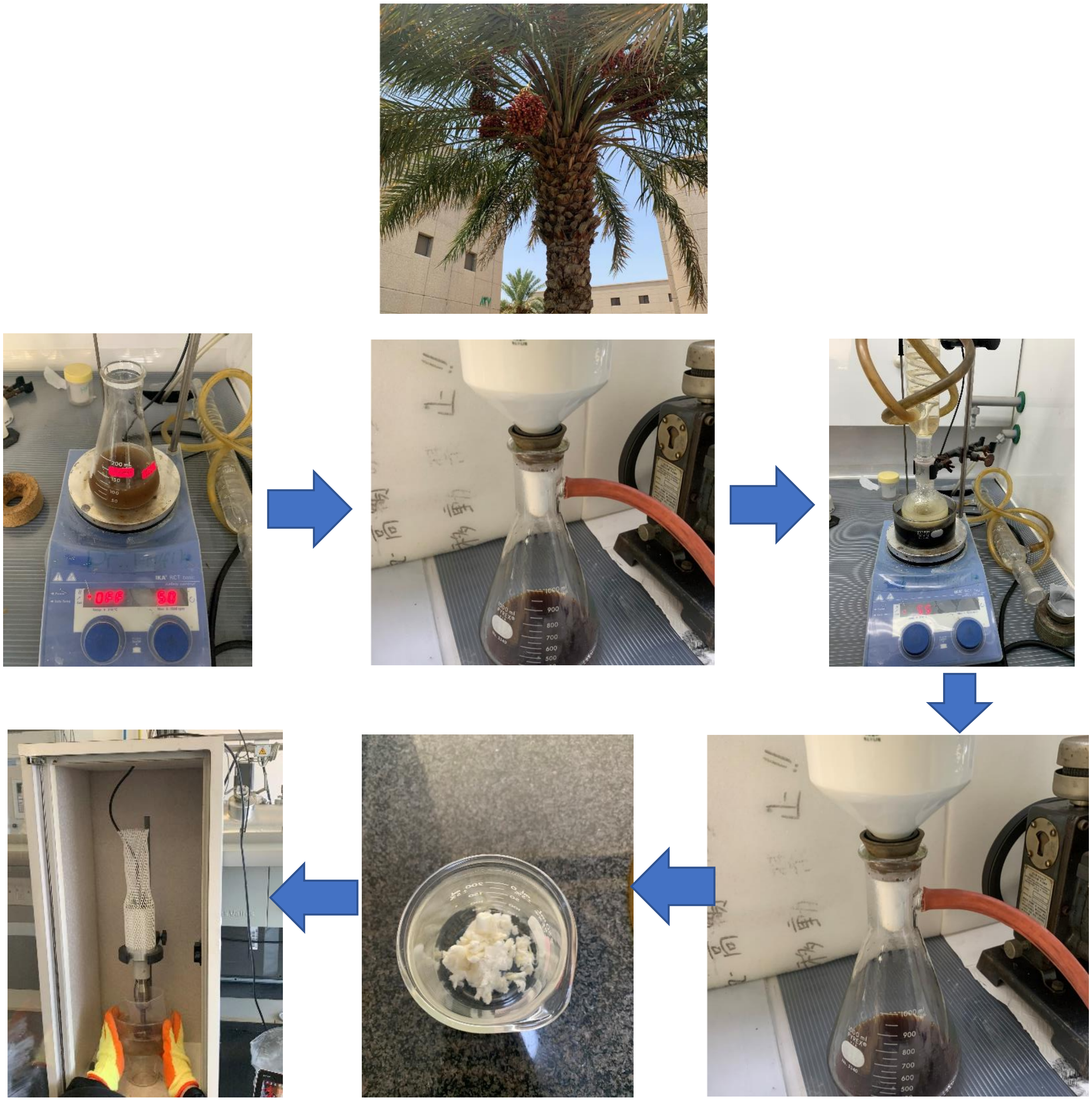


Fig 2. The flow chart of experiment

Characterization

Thermogravimetry analysis (TGA) The thermal stability of CNC/epoxy nanocomposites and epoxy Composites will be characterized using a thermogravimetric analyzer TGA STA 6000, TA Instruments at a heating rate of 15 ◦C/min under Nitrogen atmosphere (60 mL/min) from 30◦C to 700 ◦C range. The CNC sample was analyzed using X-ray diffraction (XRD) in PAN alytical X'PERT PRO MRD PW 3040/60 device at an operating voltage of 40 kV and current at 35 mA with monochromatic Cu Kα (λ=1.54 A). The intensities of diffractograms were collected in a range of $4<2\theta<45$

at a rate of 0.05°/min with a resolution of 0.25°. The morphology of nano cellulose will be observed through transmission electron microscopy (TEM) and scanning electron microscopy (SEM).

القسم ٥: النتائج

SECTION 5: RESULTS

(على سبيل المثال ، الجداول والرسوم البيانية وتحليل البيانات و/ أو الإحصائيات)

(e.g., Tables, Graphs, Data Analysis, and/or Statistics)

ضع النص الخاص بك هنا إما باللغة العربية أو الإنجليزية- إن وجدت نتائج–

As shown In Fig. 3, the TEM data showed that the particle size of nanocellulose in nano sized and form agglomeration between each other because of hydrophilic properties (strong hydrogen bonds) exhibited by this material. For SEM the result show in Fig. 5(a) for epoxy control had a clean surface. In Fig. 5(b) to 5(d) was observed the CNCs were embedded in epoxy matrix and increased with the increment of CNCs loading (from 1% to 3%). The CNCs also formed agglomeration due to strong hydrogen bonds between CNC particles within the epoxy matrix.

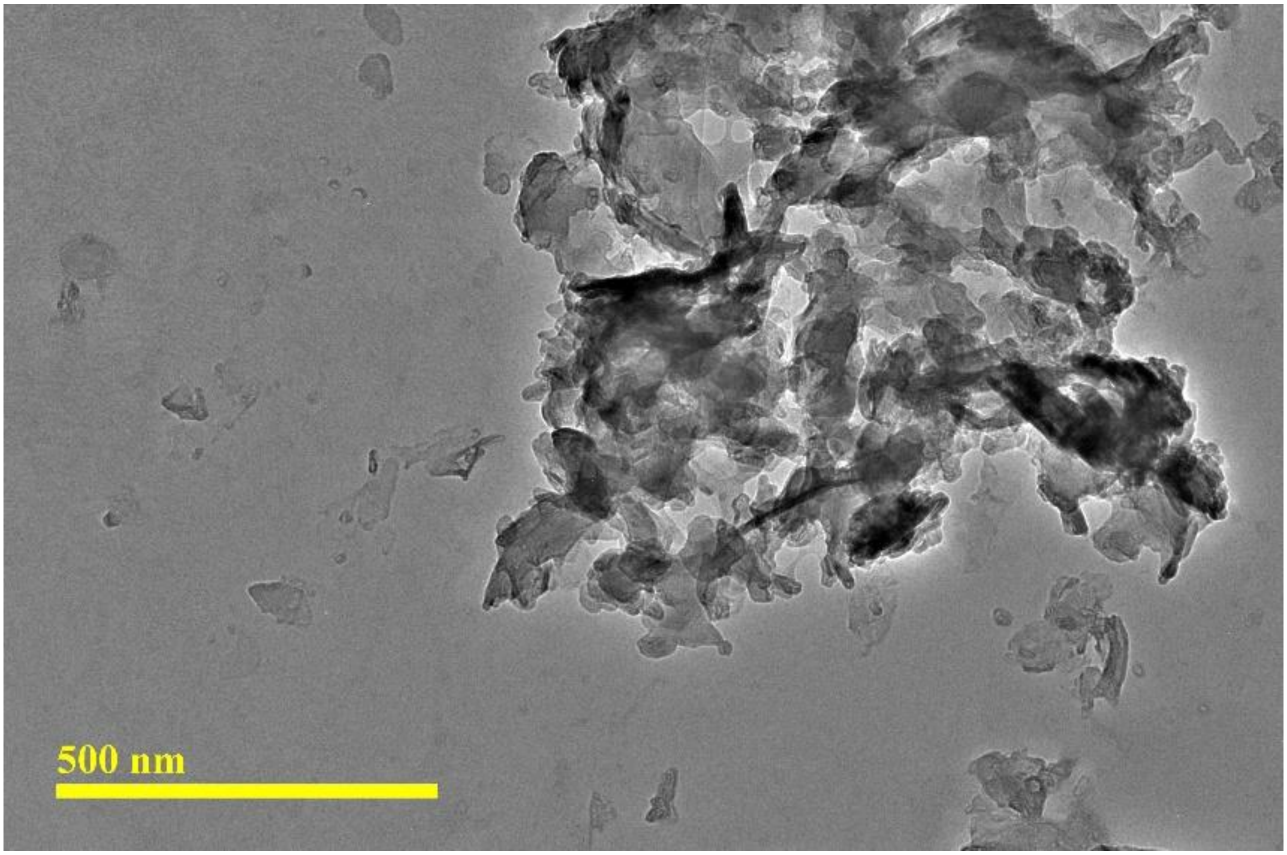
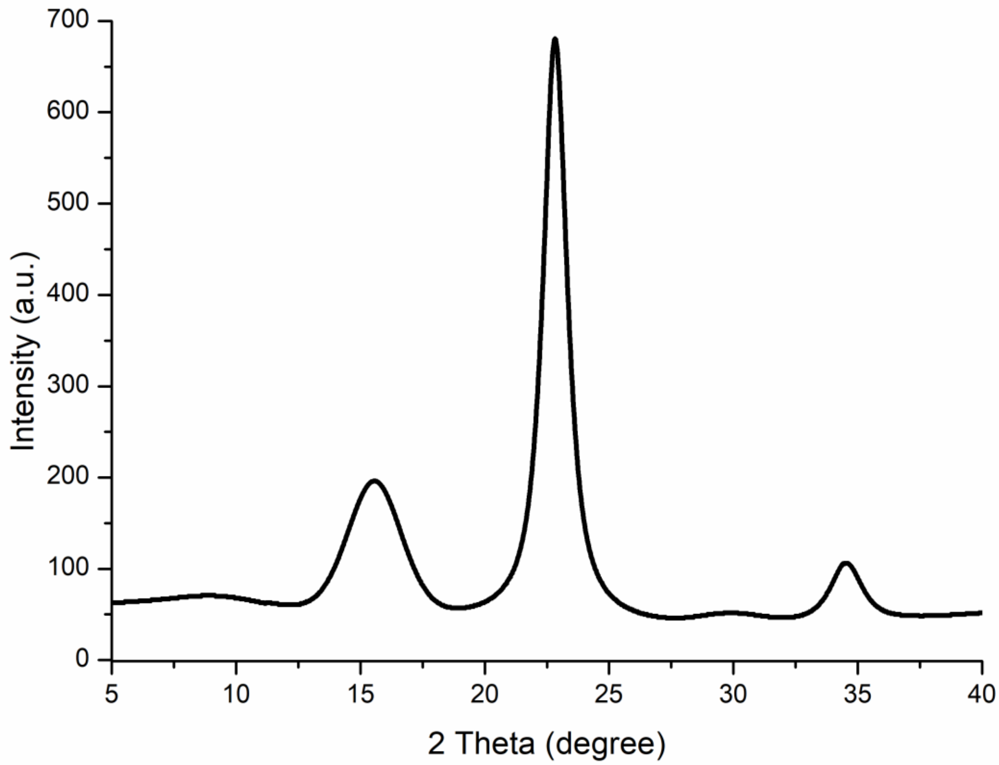


Fig. 3. TEM for nanocellulose

Fig. 4 shows the X-ray diffraction patterns for CNC extracted from date palm waste. The CNC exhibited the peaks at 2θ around 18° and 22.7°, which were attributed to the plane of (1$\bar{1}$0) and (200), respectively. This characteristic is referred to diffraction peaks for cellulose I. The CNC sample was observed with crystallinity index (CrI) of 73%. The hydrolytic cleavage of glycosidic bonds acid hydrolysis increased the crystallinity of CNC.



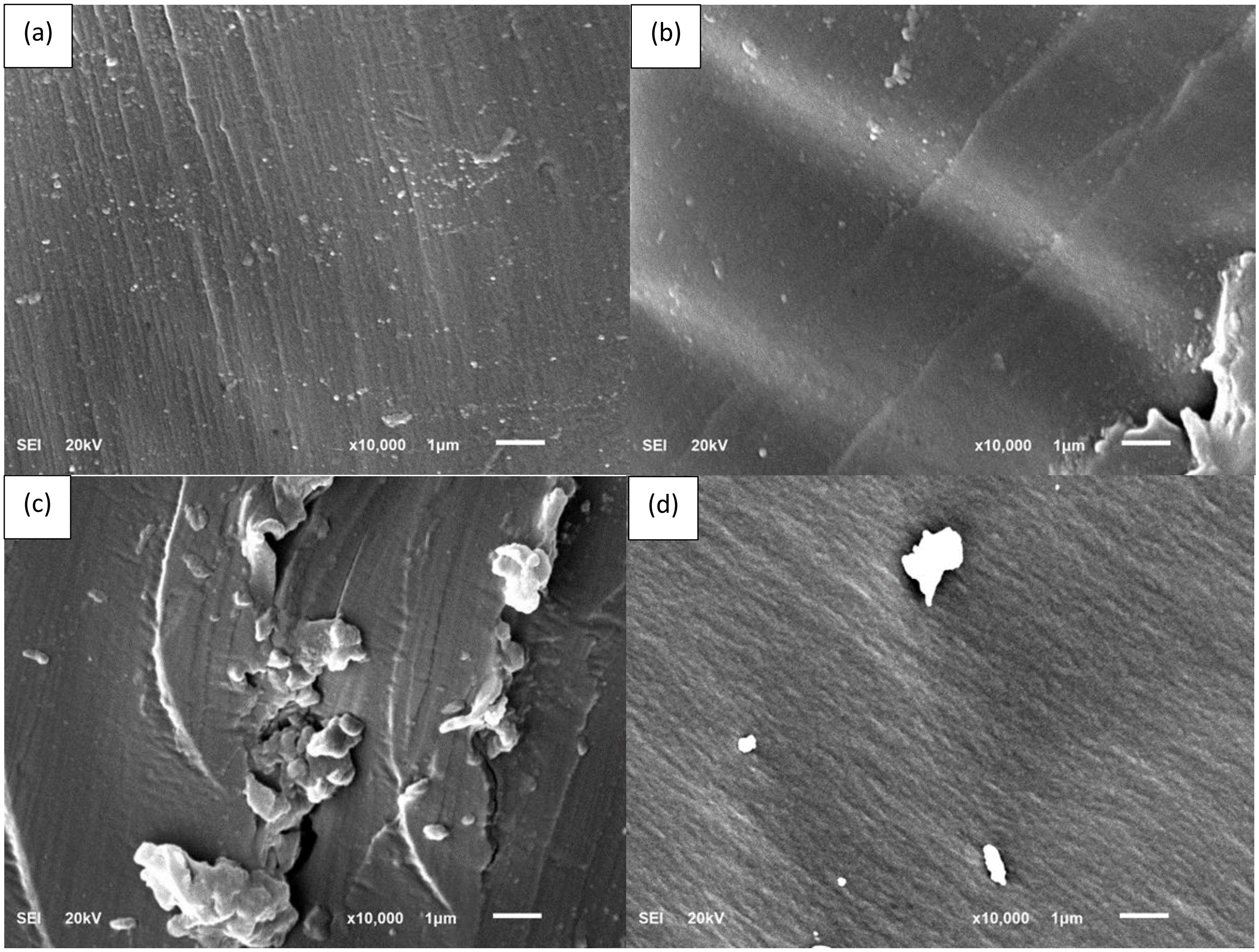


Fig. 5 Scanning electron microscopy SEM for (a) Epoxy control, (b) 1% CNC/epoxy, (c) 2% CNC/epoxy, (d) 3% CNC/epoxy

For TGA curves of epoxy control and CNC epoxy composites was observed as in Figs. 6(a) to 6(d). The $T_{10\%}$ values for epoxy control was 209 °C and for 1% CNC epoxy was 255 °C, 2% CNC epoxy was 259 °C and 3% CNC epoxy was 238 °C, respectively. that the onset degradation of CNC/epoxy matrix was observed. It was obvious why the CNC epoxy composites had higher temperature as compared than control because of addition of nanoadditives. The nanoaddtives create a strong physical bonding in epoxy polymer chains and further restricted the mobility of polymer chains. This nanoadditive also penetrated the surface pores and act as fillers. Furthermore, the high crystallinity exhibited by CNC created a barrier to heat itself. The more addition of CNC in epoxy created an agglomerated within the matrix and reduced the effectiveness to act as thermal barrier and this also reduced the uniform distribution of nanoaditives in epoxy.

DTG curves as in Figs. 6(a) to 6(d) for epoxy control values were 158 °C and 380 °C, for 1% CNC epoxy was 379 °C and 589 °C, 2% CNC epoxy 175 °C and 379 °C, 608 °C, 3% CNC epoxy 175 °C and 386 °C, respectively. the lower curve below 200 °C contributed to water evaporation and volatile components. The 1% CNC and 2% exhibited to different peaks that the first one related to degradation of CNC and later of epoxy. The addition of CNC in epoxy caused DTG curves to shift towards higher temperature and increased the thermal stability. The improvement also was contributed by formation of char that act as isolator between the bulk polymer matrix and surface when combustion occurred. The DTG curves indicate that maximum degradation temperature increased upon addition of CNC. However with a change from 1% to 3% of CNC, the T_{max} only varied a little about 7 °C as shown in DTG curves. The formation of agglomerated CNC in epoxy matrix caused the reduction in thermal stability for 3% CNC epoxy.

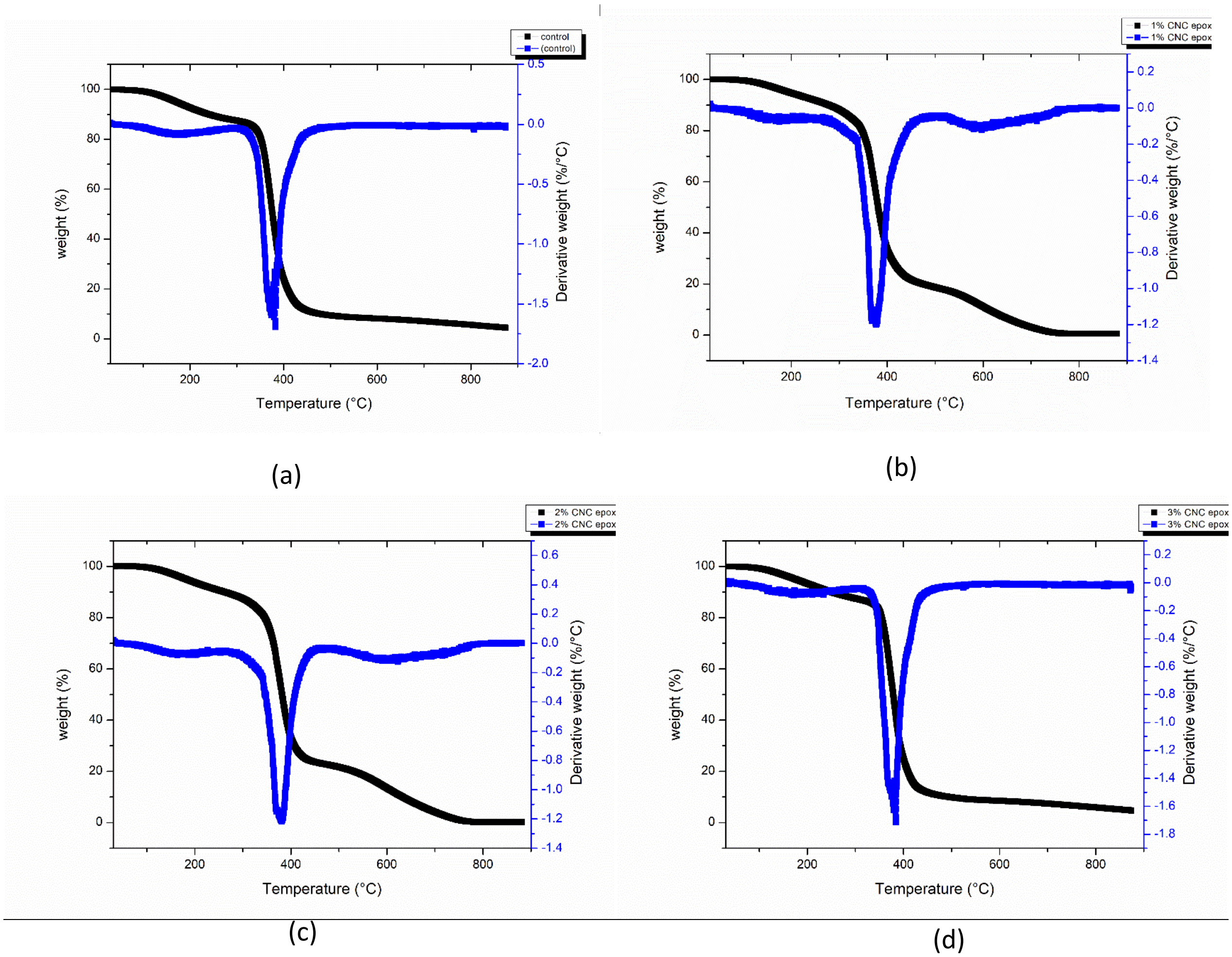


Fig. 6 TGA and DTG for (a) epoxy control, (b) 1% CNC epoxy, (c) 2% CNC epoxy, and (d) 3% CNC epoxy

القسم ٦: التفسير والاستنتاجات

SECTION 6: INTERPRETATION & CONCLUSIONS

(على سبيل المثال ، المساهمات والقيمة المضافة والتأثير و/أو التطبيقات)

(e.g., Contributions, Value Add, Impact, and/or Applications)

ضع النص الخاص بك هنا إما باللغة العربية أو الإنجليزية- إن وجدت نتائج-

Conclusion:

In a nutshell, the epoxy composite with different loadings (1-3%) of nanocellulose was successfully fabricated and the thermal stability was improved. The results showed that improvement of this thermal stability was achieved with a small amount of 2% CNC addition of nanocellulose in epoxy matrix and improve for 23.9% higher as compared than control sample. Further addition of nanocellulose with 1% wasn't effective enough in improving thermal stability, and the addition of 3% decreased the effectiveness in thermal stability because of agglomeration occurred between CNC particles due to strong hydrogen bonding.

Applications:

It can be utilized for advanced material applications where thermal stability along with renewability , as heat insulator , and for different applications such as electronics parts, steering wheels, door panel, floor panel, car seats and so on…

Future work:

For the short term is to added nano cellulose into polymer, namely epoxy and investigate thermal stability and morphological study, and in future we can do investigate Mechanical, biodegradable, and physical properties of this nano cellulose epoxy composite as well try to mix with different polymer type such as poly(vinyl alcohol), poly(vinyl chloride), poly propylene (PP) and elastomers.

القسم ٧: المراجع

SECTION 7: REFERENCES

ضع قائمة بأهم المراجع (كتب ، مقالات علمية، ...)

1. Rezanova V, Rezanova N, Plavan V, Viltsaniuk O. The influence of nano-additives on the formation of matrix-fibrillar structure in the polymer mixture melts and on the properties of complex threads. Fibres and Textiles. 2017.
2. Huang G, Chen W, Wu T, Guo H, Fu C, Xue Y, et al. Multifunctional graphene-based nano-additives toward high-performance polymer nanocomposites with enhanced mechanical, thermal, flame retardancy and smoke suppressive properties. Chemical Engineering Journal. 2021;410:127590.
3. Shifrina ZB, Matveeva VG, Bronstein LM. Role of polymer structures in catalysis by transition metal and metal oxide nanoparticle composites. Chemical reviews. 2019;120(2):1350-96.
4. Börjesson M, Westman G. Crystalline nanocellulose—preparation, modification, and properties. Cellulose-fundamental aspects and current trends. 2015;7.
5. Isogai A. Emerging Nanocellulose Technologies: Recent Developments. Advanced Materials. 2021;33(28):2000630.
6. Phanthong P, Reubroycharoen P, Hao X, Xu G, Abudula A, Guan G. Nanocellulose: Extraction and application. Carbon Resources Conversion. 2018;1(1):32-43.
7. Gan PG, Sam ST, Abdullah MFb, Omar MF. Thermal properties of nanocellulose-reinforced composites: A review. Journal of Applied Polymer Science. 2020;137(11):48544.
8. Zabihi O, Ghasemlou S. Nano-CuO/epoxy composites: thermal characterization and thermo-oxidative degradation. International Journal of Polymer Analysis and Characterization. 2012;17(2):108-21.
9. Nishitsuji DA, Marinucci G, Evora MC, e Silva LGdA. Study of electron beam curing process using epoxy resin system. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms. 2007;265(1):135-8.
10. Ghasemi-Kahrizsangi A, Neshati J, Shariatpanahi H, Akbarinezhad E. Improving the UV degradation resistance of epoxy coatings using modified carbon black nanoparticles. Progress in Organic Coatings. 2015;85:199-207.
11. Chakrabarty A, Teramoto Y. Recent advances in nanocellulose composites with polymers: A guide for choosing partners and how to incorporate them. Polymers. 2018;10(5):517.
12. Dufresne A. Nanocellulose Processing Properties and Potential Applications. Current Forestry Reports. 2019;5(2):76-89.
13. Yahya SA, Iqbal T, Omar MM, Ahmad M. Techno-economic analysis of fast pyrolysis of date palm waste for adoption in Saudi Arabia. Energies. 2021;14(19):6048.
14. Hussain A, Farooq A, Bassyouni MI, Sait HH, El-Wafa MA, Hasan SW, et al. Pyrolysis of Saudi Arabian date palm waste: A viable option for converting waste into wealth. 2014.
15. Faiad A, Alsmari M, Ahmed MM, Bouazizi ML, Alzahrani B, Alrobei H. Date palm tree waste recycling: treatment and processing for potential engineering applications. Sustainability. 2022;14(3):1134.
16. Morais JPS, de Freitas Rosa M, Nascimento LD, Do Nascimento DM, Cassales AR. Extraction and characterization of nanocellulose structures from raw cotton linter. Carbohydrate polymers. 2013;91(1):229-35.
17. Csiszar E, Kalic P, Kobol A, de Paulo Ferreira E. The effect of low frequency ultrasound on the production and properties of nanocrystalline cellulose suspensions and films. Ultrasonics Sonochemistry. 2016;31:473-80.
18. Jamaluddin N, Kanno T, Asoh T-A, Uyama H. Surface modification of cellulose nanofiber using acid anhydride for poly (lactic acid) reinforcement. Materials Today Communications. 2019;21:100587.


good luck
to you

【評語】030034

Nanocellulose is an interesting material that is transparent and has good mechanical strength. This work investigates a composite based on epoxy and nanocellulose from palm waste. The presentation is clear and concise.

The authors tried to incorporate nano additives into epoxy as a polymer matrix to enhance and improve the thermal stability of composite by crosslinking the polymer chains with the nano additives extracted from date palm waste to create environmentally friendly and sustainable nano additives products. They have used XRD, SEM, and TGA/DTG to characterize the resulting materials. The nano additives create a strong physical bonding in epoxy polymer chains and further restrict the mobility of polymer chains. The nano additives can also penetrate the surface pores and act as fillers. Furthermore, the high crystallinity exhibited by crystalline nanocellulose (CNC) created a barrier to heat itself. The work is interesting and may be utilized as a heat insulator for applications in electronics parts, steering wheels, door and floor panels, car seats, etc.