



# **iJRASET**

International Journal For Research in  
Applied Science and Engineering Technology



---

# **INTERNATIONAL JOURNAL FOR RESEARCH**

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

---

**Volume: 7      Issue: IV      Month of publication: April 2019**

**DOI: <https://doi.org/10.22214/ijraset.2019.4336>**

**[www.ijraset.com](http://www.ijraset.com)**

**Call:  08813907089**

**E-mail ID: [ijraset@gmail.com](mailto:ijraset@gmail.com)**

# Bio-Mass Material as a Source of Micro-crystalline Cellulose by Strong Acid Treatments.

Ahmad Mousa<sup>1</sup>, M. Al-Edwan<sup>2</sup>, A. Khresat<sup>3</sup>, S. Al-Zoubi<sup>4</sup>, Z. Yazjeen<sup>5</sup>

<sup>1, 2, 3, 4, 5</sup>Department of Materials Engineering, Faculty of Engineering, Al Balqa Applied University, Salt 19117 Jordan

**Abstract:** Due to environmental concerns agro-waste material namely wheat straw (WS) will be utilized to produce microcrystalline cellulose (MCC) via mercerization, bleaching and strong acid hydrolysis. The current investigation compared MCC produced by phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and hydrochloric acid (HCL). The percentage yield of each process were compared and found to be the highest for the H<sub>3</sub>PO<sub>4</sub> compared H<sub>2</sub>SO<sub>4</sub> and HCL. The morphology of the three products was viewed under scanning electron microscope (SEM). The heat of dissolution was studied for the three samples, and found to be the highest for the H<sub>3</sub>PO<sub>4</sub>, which is in line with %yield. This is also indicative that the chemical attack on the amorphous regions was the highest for H<sub>3</sub>PO<sub>4</sub>.

## I. INTRODUCTION

Manufacturing of high performance engineering materials such as micro crystalline cellulose (MCC) from renewable resources such as agro-waste materials is one of ambitious goals currently being pursued by researchers all over the world. MCC is a biodegradable material which is of porous particle texture. The MCC find its way to applied in industries such as pharmaceutical, cosmetics and food industries as well as water retainer and reinforcement filler in bio-composite fabrication.

Due to the increasing concern about the environment and CO<sub>2</sub> emissions, the scientific community have considered the agricultural wastes such as coconut husk fibers, groundnut husk, rice straw and rice husk to produce micro crystalline cellulose (1-5 ). The choice of the agro-waste is related to geographical regions, in other words to what is common and abundant in the region. Referring to agro-waste materials in Jordan, wheat straw (WS) and olive pomace (OP) are two common agricultural wastes they were used as building material, burned in the open atmosphere which produces a lot of CO<sub>2</sub>, further it is being used to feed animals especially the wheat straw. However, recently various attempts were made by us to produce MCC from olive pomace (6-18 ), but up to our knowledge no attempt has been made to extract MCC from the WS and. In this work MCC will be extracted from wheat straw (WS) by the removal of non-cellulosic components relying on mercerization treatment of the WS followed by hydrolysis technique.

## II. EXPERIMENTAL

### A. Materials

WS was collected from a local wheat farm in Jordan. Sodium hypochlorite (NaOCl) and sodium hydroxide (NaOH) were used as bleaching agents while H<sub>3</sub>PO<sub>4</sub>, H<sub>2</sub>SO<sub>4</sub> and HCl were used for acid hydrolysis process. All chemical were used without further purification.

### B. Alkaline and Bleaching Treatments of WS

The received WS was washed with distilled water and dried under air for fourteen days followed by drying using air circulating at 100 °C. The dried sample was ball milled followed by sieving on a Retsch AS 200 shaker for ten minutes. WS powder was treated with 10% aqueous sodium hydroxide (NaOH) solution at room temperature, maintaining a liquor ratio of 20:1 for 24h to remove the hemicelluloses and other greasy materials. The solution was filtered and the residue washed with distilled water for removal of solubilized components until pH 7 was achieved. The alkaline treated WS then was undergone bleaching treatment with 5% NaOCl for 6h at room temperature. The resulted cellulose obtained from the treated WS was filtered and rinsed several times using distilled water. It was dried in an oven until constant weight was recorded. The dried powder was grounded into fine powder using rotary ball mill (19-22).

### C. Acid Hydrolysis

The obtained cellulose was hydrolyzed using 5% acid solution at room temperature under continuous stirring. The MCC collected was filtered and rinsed with distilled water until pH 7 was achieved followed by air circulating oven drying until constant weight was obtained. The % yield of the MCC calculated according to equation 1 (16) is reported in table 1.

$$x = \frac{B}{A} * 100 \quad (1)$$

Where B is the final weight in grams and A is the initial weight.

### III. RESULTS AND DISCUSSIONS

#### A. Percentage Yield of MCC

The effect of acid hydrolysis on the % yield of WS is reflected in Table 1.

| ingredient(g)     | W1(g) | W2(g) | % Yield |
|-------------------|-------|-------|---------|
| Ethanol           | 100   | 90    | 90      |
| Sodium hydroxide  | 90    | 70    | 78      |
| Hypochlorite      | 70    | 60    | 86      |
| Hydrochloric acid | 20    | 15    | 75      |
| sulfuric acid     | 20    | 13    | 65      |
| phosphoric acid   | 20    | 14    | 70      |

Table1: percentage yield of MCC extracted by various strong acids treatments.

In this regard the mercerized samples have been exposed to three different acids. These were  $H_3PO_4$ ,  $H_2SO_4$  and  $HCl$  at 5% concentration. The rationale behind the acid treatment is based on the fact that the hydronium ions of the mineral acids are capable to penetrate and remove the excessive amorphous regions of cellulose; this fact was adopted and used to explain the obtained results in this study. Note that the amount of MCC obtained after the  $HCl$  treatment was the highest followed by  $H_3PO_4$ ,  $H_2SO_4$  respectively. The different yield% should be correlated to the solvating power of each acid. It is noted that sulfuric acid treatment has come out with the less amount of % yield; this is a hint that the sulfuric acid has the highest solvating power toward the WS compared to the other two acids. This is a remark that the sulfuric acid was able to access the WS and to dissolve higher amounts of the amorphous regions within the WS; hence lower % yield was detected but more crystalline product. To further support this tend the heat of dissolution was estimated using bomb according to equation 2. The results of the calorimetry study is shown in table 2.

$$Q = cm\Delta T \quad (2)$$

Where:  $Q$  is the heat released,  $c$ , is the specific heat,  $m$  is the mass, and  $\Delta T$  is the change in temperature. Note that the heat of dissolution ( $Q$ ) is the highest in case of sulfuric acid treatments compared to the other two acids. This is again confirms our claim that the sulfuric acid has the highest solvation power, in other words it has dissolved the maximum amount of amorphous cellulose, consequently the least %yield.

| Chemical Agent | Q1 (kj/g) | Q2(kj/g) | $\Delta Q(kj/g)$ |
|----------------|-----------|----------|------------------|
| HCl            | 3         | 2        | 1                |
| $H_3PO_4$      | 8         | 4        | 4                |
| $H_2SO_4$      | 51        | 34       | 17               |

Table 2: shows the heat of enthalpy for the reactions with the wheat straw



### B. Scanning Electron Microscope Study

Figure 1 shows the SEM micrographs of the raw and hydrolyzed biomass. The microstructure of these samples was examined to understand the influence of chemical treatment on the structural changes, particle size and shape of the biomass powder. It can be seen that the raw material (as received) is a non-porous solid mass.

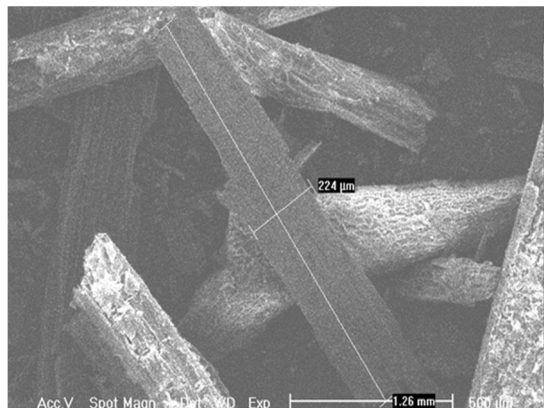


Fig.1: SEM images of the as received WS.

| Sample                                 | Width (μm) |
|----------------------------------------|------------|
| Raw sample                             | 224        |
| HCl treated                            | 31.5       |
| H <sub>3</sub> PO <sub>4</sub> Treated | 42.5       |
| H <sub>2</sub> SO <sub>4</sub> Treated | 41.5       |

Table3: The effect of various acid treatments on the particle size of the WS.

Furthermore, it is noted that the material is not isometric with fiber like or agglomerates bundled together with high diameter as evidenced from the scanning electron microscope images displayed in Figure 1. In this regard the influence of the acid treatment on the particle size of the WS is compared in table 3. In term of particle dimensions, it is noted that external fiber diameter (width) is 224 μm. The effect of acid treatment on the alkali bleached WS sample is show in in Figures 2-4 consequently. Figure 2 displayed the microstructure of the hydrochloric acid treated sample. The aim is to compare its potential to release the crystalline regions with other acid potentials.

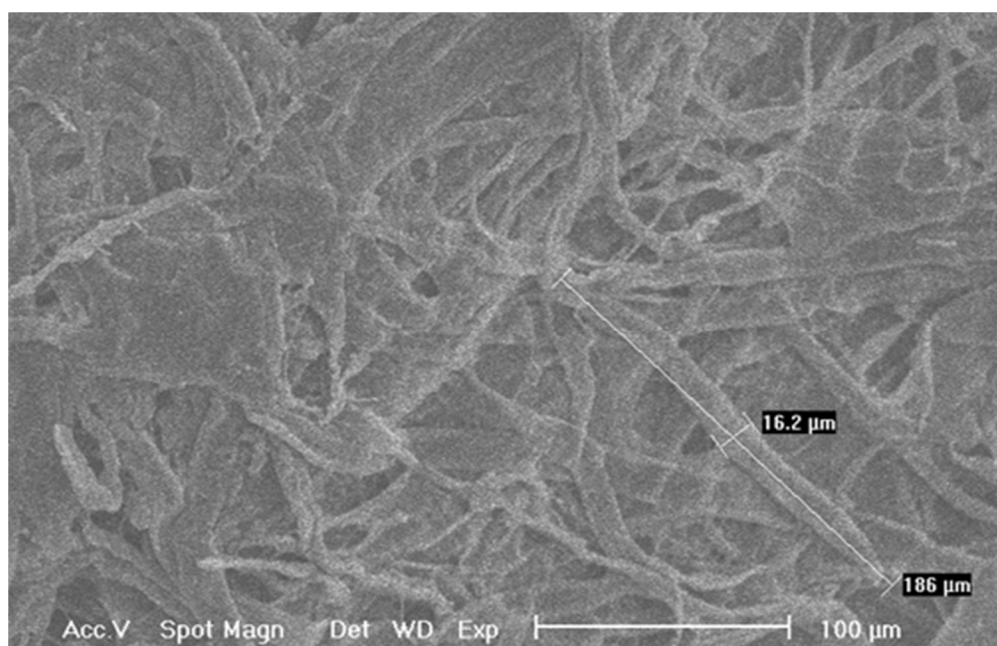


Fig.3: SEM images of the Phosphoric acid hydrolyzed WS.

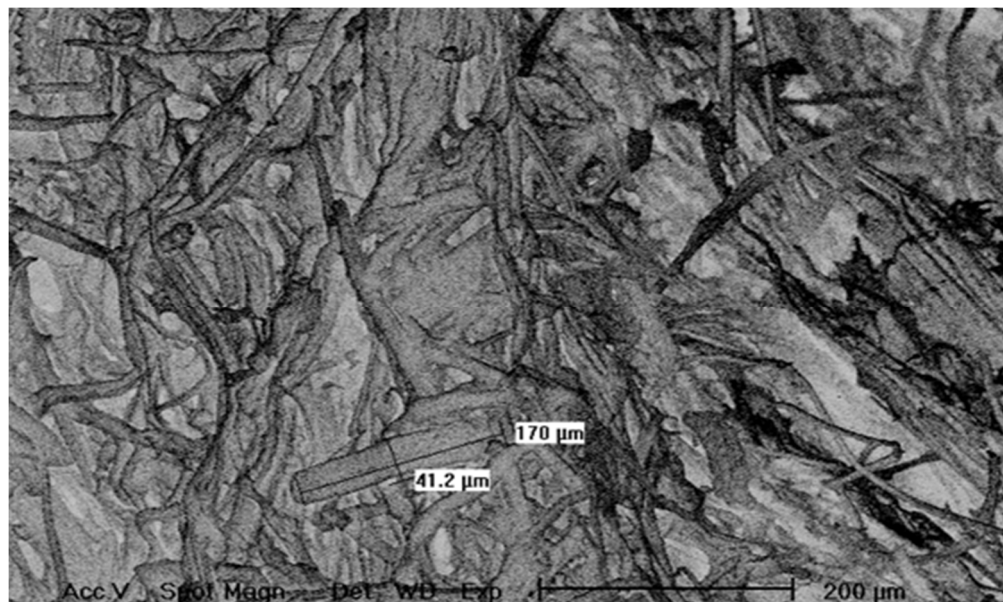


Fig.4: SEM images of the sulfuric acid hydrolyzed WS.

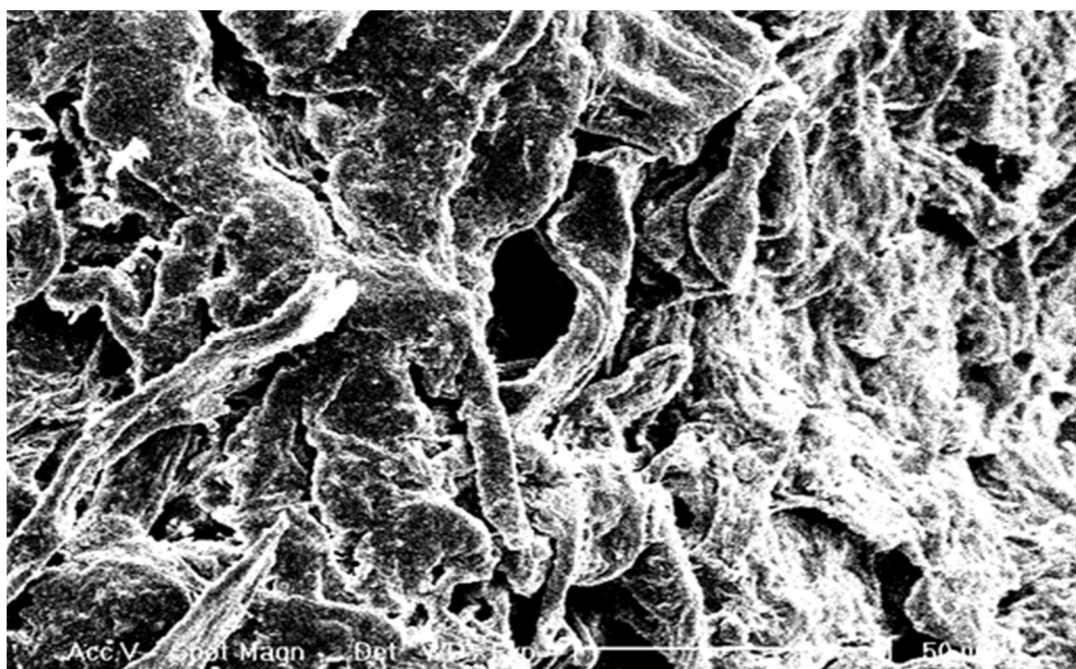


Fig.5 : SEM images of the hydrochloric acid hydrolyzed WS.



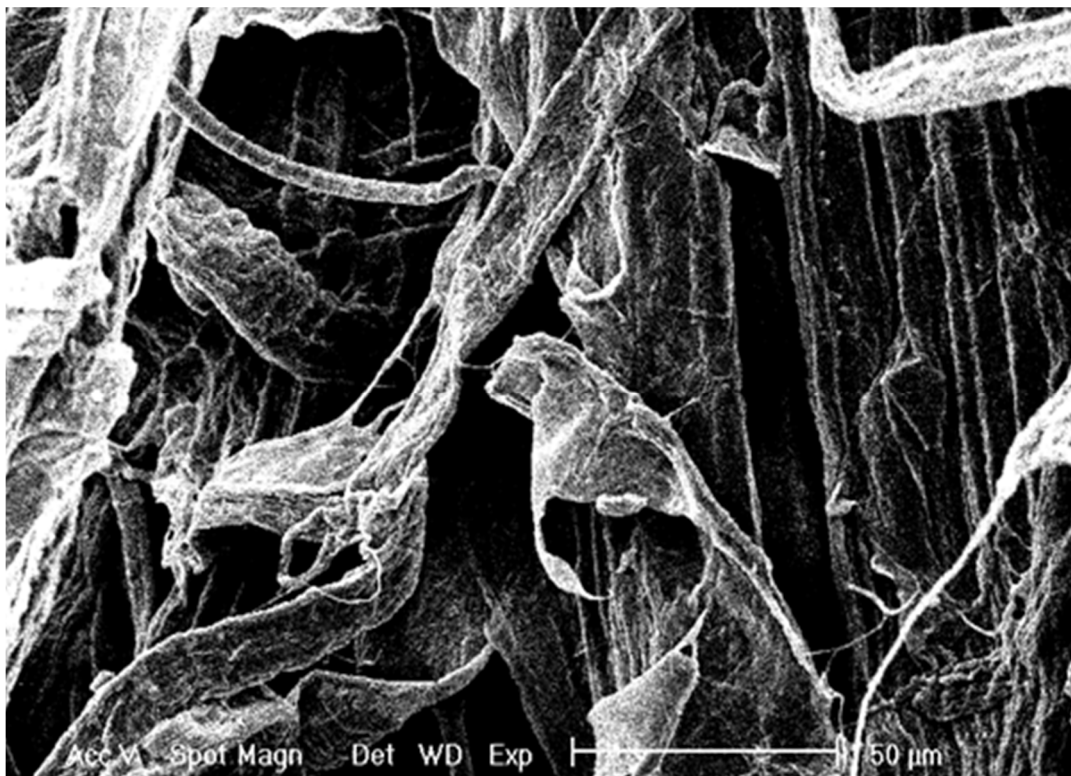


Fig.6: SEM images of the phosphoric acid hydrolyzed WS

#### IV. CONCLUSION

Based on the aforementioned results, the following facts can be concluded

- A. The alkali treatment followed by acid hydrolysis was able to dissolve the non-cellulosic components and to release the MCC-WS.
- B. The sulfuric acid based hydrolysis displayed the best results compared to the other two acidic treatments. This was evidenced by the higher aspect ratio of the MCC shown in table3 and the improved surface area shown by SEM images.

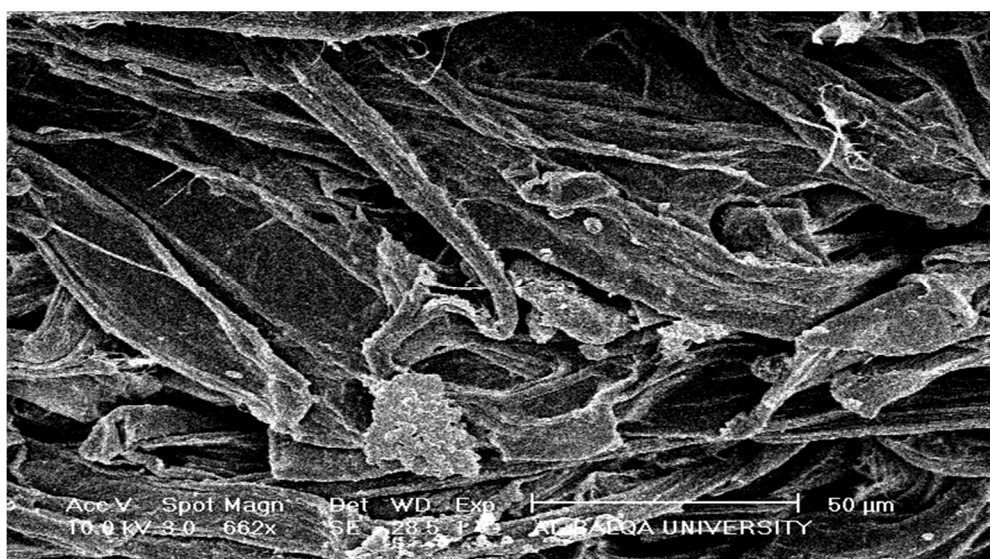


Fig.7: SEM images of the sulfuric acid hydrolyzed WS.

It is clear that the hydrochloric acid treatment has tremendously influenced the surface topography. Note that surface texture has been converted to thinner fiber like material as indicated by the lower diameter ( $31.5\mu\text{m}$ ). On the other hand the phosphoric acid treated material shown in Figure 3 showed further improvements in the sense that more dimensional reduction has occurred compared to that of hydrochloric acid treatments. This should be attributed to the solvating power of the acid and its capability to extract the amorphous regions. Figure 4 demonstrates the influence of sulfuric acid treatment on the microstructure of the WS compared to the other acidic treatments. It is clear that further reduction in the dimension were obtained, this is again should be related to the removal of the amorphous regions. As a result of the particle size reduction due to the breakdown of the cellulose molecules, one can expect that such dimensional change was accompanied by an increase in the surface area of the treated WS materials, remember that that the smaller the particle size the higher the surface area is. Furthermore, the enhanced surface area could be indirectly quantified from the SEM micrographs as shown in 5-7, consequently. Apparently the surface appearance was converted to flattened shape with respect to the raw sample shown in Figure 1.

## REFERENCES

- [1] Mousa, Heinrich , Udo Wagenknecht, Toughened unsaturated polyester composites reinforced with landfill Material, polymer plastic technology and engineering, 2017
- [2] Mousa, Gert . Heinrich , Udo Wagenknecht , Characterization of dynamically vulcanized PS/SBR composites filled exfoliated graphite the Journal of Engineering Materials and Technology, 2017
- [3] Mousa and G. Heinrich, Different Characterization of solid Bio-fillers based agricultural waste material. "Handbook of Composite from Renewable Materials" Volume 3: Physico-chemical and Mechanical Characterization" being published by Wiley-Scrivener, to appear , Dec 2016.
- [4] Mousa, G. Heinrich, U. Wagenknecht & D. Jehnichen: Utilization of cellulose based agro-waste as reinforcement for unsaturated polyester composites, Int J Plast Technol (2016)
- [5] Mousa, G. Heinrich, U. Wagenknecht. Wood like material from thermoplastic polymer and Landfill Bio-Materials: DMA, TGA and solvent resistance properties Polymers from renewable materials 6 (2015) 25-42.
- [6] Mousa, G. Heinrich, U. Wagenknecht, Bio-Based Fillers, Encyclopedia of Polymeric Nanomaterials, Springer (2015) 106-109,
- [7] Mousa, G. Heinrich, U. Wagenknecht, Wood like material from thermoplastic polymer and Landfill Bio-Materials: Water absorption, Thermal and Morphological studies' Polymers from Renewable Resources, 5 (2014) 1-18
- [8] Mousa, G. Heinrich, U. Wagenknecht, Thermal properties of carboxylated nitrile rubber/nylon-12 composites-filled lignocellulose materials : J. of Thermoplastic Composite Materials, 27 (2014) 167-179
- [9] A. Mousa, G. Heinrich, U. Wagenknecht, THE APPLICATION OF SOLID OLIVE WASTE AS REINFORCEMENT IN CARBOXYLATED NITRILE BUTADIENE RUBBER /ORGANO NANO LAYERED SILICATES COMPOSITES, J. Of Solid waste Technology and Managemnet, 39(3), 2013
- [10] A. Mousa , G. Heinrich, The Effect of Microwave Irradiation on the Physical and Morphological Behavior of Olive Husk Biomass and its Application in XNBR Vulcanizates Biomass and waste valorization, : (2012) 3:157-164, DOI 10.1007/s12649-011-9106-2
- [11] Mousa, G. Heinrich , Bernd Kretschmar, U. Wagenknecht, Amit Das. Utilization of Agrowaste Polymers in PVC/NBR Alloys: Tensile, Thermal and Morphological Properties international J. Of chemical Engineering, Volume 12, Article ID 121496, 5 pages doi:10.1155/2012/121496
- [12] Mousa, G. Heinrich , The Effect of Silane Treated Hybrid Filler on the Mechanical and Thermal Performance of Carboxylated Nitrile Butadiene Rubber (XNBR) Composites . International Polymer processing, (2012) 3; page 1–6: DOI 10.3139/217.2522.
- [13] Mousa, G. Heinrich. The Application of Di-isocyanate Modified Agro-polymer as Filler For XNBR/PA-12 Thermoplastic Elastomer Composites. Macromolecular science part A: Pure and Applied chemistry, 2012 , 49:5, 385-396
- [14] Mousa , G. Heinrich , U. Wagenknecht : Rubber-Wood Composites from Chemically Modified Olive Husk Powder and Carboxylated Nitrile Butadiene Rubber: Cure Characteristics, Tensile Behavior, and Morphological Studies: Journal of Wood Chemistry and Technology, 32:82–92, 2012: DOI: 10.1080/02773813.2011.599469
- [15] A Mousa, G Heinrich, F Simon, U Wagenknecht, Carboxylated nitrile butadiene rubber/hybrid filler composites - Materials Research, 2012
- [16] Mousa, G. Heinrich, U. Wagenknecht, U. Gohs. Mechanical Properties of Electron Beam Treated Carboxylated Nitrile Butadiene Rubber (XNBR) composites reinforced by Organic/inorganic Hybrid Filler J. of Composites Materials , 46(10) 1151–1157(2012).
- [17] Mousa, G. Heinrich. Cure Characteristics and Mechanical Properties of Carboxylated Nitrile Butadiene Rubber (XNBR) vulcanizate reinforced by Organic Filler. Polymers-Plastics Technology and Engineering, 2011: 50:13, 1388-1392 <http://dx.doi.org/10.1080/03602559.2011.584242>
- [18] Mousa, G. Heinrich and U. Wagenknecht, Thermoplastic Composites Based on Renewable Natural Resources :Unplasticized PVC/Olive Husk, International J. of Polymeric Materials, 2010, 59:843–853: DOI: 10.1080/00914037.2010.504143
- [19] Mousa, G. Heinrich, U. Gohs , R. Hassler and U. Wagenknecht "Application of Renewable Agro-waste Based Olive Pomace on the Mechanical and Thermal Performance of Toughened PVC. Polymers-Plastics Technology and Engineering J; 48,1030-1040, 2009.
- [20] Mousa, Evolution of Mechanical Properties of EPDM Vulcanizates by compounding with layered Organo-Montmorillinite, International Journal of Polymeric Materials, 56, 4 (2007). 355–363
- [21] Mousa, Cure Characteristics and Thermal Properties of Sulfur Cured EPDM Based Composites by Compounding with Layered Nano-Organoclays, Polymers-Plastics Technology and Engineering, 45, 8 (2006) 911-915.
- [22] Karger-Kocsis, J Gremmels, A Mousa, US Ishaku, ZA Mohd Ishak, Application of hygrothermally decomposed polyurethane in rubber recipes. Part 1: Natural rubber (NR) and nitrile rubber (NBR) stocks, - GK Kautschuk Gummi Kunststoffe, 528-533, 2000
- [23] A Mousa, US Ishaku, ZA Mohd Ishak - Plastics rubber and composites processing , Dynamic vulcanisation of poly (vinyl chloride)-epoxidised natural rubber thermoplastic elastomers. Part 1-Mixing rheology, 26(8), 331-335 1997



[24] A Mousa, G Heinrich, F Simon, U Wagenknecht, Carboxylated nitrile butadiene rubber/hybrid filler composites - Materials Research, 2012





10.22214/IJRASET



45.98



IMPACT FACTOR:  
7.129



IMPACT FACTOR:  
7.429



# INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Call : 08813907089  (24\*7 Support on Whatsapp)