

Comparison of different Poly vinyl chloride (PVC) /Calcium Carbonate (CaCO_3) blends and their properties

Kardo Khalid Abdullah^{1*}, Kálmán Marossy², Kinga Tamási³

¹Faculty of Materials Science and Engineering / Institute of Ceramic and Polymer Engineering - University of Miskolc

²Professor, Institute of Ceramic and Polymer Engineering - University of Miskolc

³PhD Student, Institute of Ceramic and Polymer Engineering - University of Miskolc

*Corresponding Author

Received: 10 September 2021/ Revised: 18 September 2021/ Accepted: 24 September 2021/ Published: 30-09-2021

Copyright © 2021 International Journal of Engineering Research and Science

This is an Open-Access article distributed under the terms of the Creative Commons Attribution

Non-Commercial License (<https://creativecommons.org/licenses/by-nc/4.0>) which permits unrestricted

Non-commercial use, distribution, and reproduction in any medium, provided the original work is properly cited.

Abstract— The effects of micro and nanoscale calcium carbonate (CaCO_3) particles on the mechanical properties of polyvinyl chloride (PVC) were investigated. Type of (PVC S5070) were used as the matrix in this study, hydrocarb 95T CaCO_3 as a micro size particles, and SOCAL 312 CaCO_3 as a nano size particles with different phr (0 wt%, 10 wt% and 20 wt%). The nano- CaCO_3 particles were observed by FT-IR spectras. Nano- CaCO_3 increased the tensile strength and affected the tensile strength of PVC more than micro- CaCO_3 particles. The raise in hardness shore (A) is more considerable in nano- CaCO_3 filled PVC compared to that of micro- CaCO_3 . TSDC test investigated that nano- CaCO_3 in PVC samples have greater electrical resistance, and the maximum volume resistivity in micro- CaCO_3 in PVC samples which mixed by ultrasonic. Thermal characterization of PVC samples were tested by DMA. Storage modulus and loss factor ($\tan \delta$) examined and showed that the storage modulus of PVC decreased as a function of temperature. The storage modulus can be increased concurrently by the presence of CaCO_3 . The glass transition of nano- CaCO_3 sample mixed by ultrasonic has higher glass transition than other samples which mixed by normal mixing.

Keywords— Calcium carbonate, Polymer composites, PVC, Micro- CaCO_3 , Nano- CaCO_3 .

I. INTRODUCTION

Poly (vinyl chloride) as well known as PVC is one of the most using polymers around the world because of being in low cost [1-4], durability [5-7], flexibility [8-10]. However, they need to enhance their mechanical properties [11-13] such as toughness, morphological, thermal, and processing properties to work properly in widely and different range fields [14-17]. The mechanical properties of blends change as per various fillers [18]. Much improvement of the poorer properties of PVC has been carried out by the combination of additives such as plasticizers [19-20], antioxidant [21], and flame-retardant fillers [22-24] higher thermal stability [25] copolymerization, fillers and heat stabilizers with other monomers. Calcium carbonate (CaCO_3) nanoparticles are one of the viable nanofillers that enhance the thermo-mechanical properties of the polymer matrix composites [26-28].

PVC is the most common used to produce pipes, windows, entryways, and others in the development business [29-34]. The utilization of nanoparticles of calcium carbonate, not just improves the relentlessness, but also enhances electrical properties, heat opposition, radiation obstruction and different properties [35-39], yet in addition lessens the expense of the mixes [40-43], also in medical uses PVC have roles [44-48]. The mechanical properties of CaCO_3 microstructural additive on PVC are totally dependent on the interaction between the fillers [49-51], the polymeric matrix and the filling ratio, and the surface attributes [52-56].

The effects of the loads on the microstructure and on the properties of the PVC compounds are depending on the magnitude size and shape of the particle, combined size, surface treatment of the polymer and CaCO_3 filler. Therefore, by adding some low-cost additives to the PVC to develop it and create new formulation, it will be more affective and widely uses in more ways and application [57-59]. So as to acquire higher been utilized to treat nano CaCO_3 particles to improve the interfacial bond between the PVC lattice and the nano CaCO_3 molecule. The most regularly utilized adjusting operators

to treat nano CaCO_3 molecule are stearate and titanate coupling specialists, and the last is noted to have a higher hardening and strengthening impact [60].

It is notable that calcium carbonate (CaCO_3) is a characteristic happening and bountiful mineral involving around 4% of earth crust [61-63]. Lately, many tests and exams have been committed to the calcium carbonate particles [64-67]. By adding some amount of calcium carbonate, tensile strength and impact strength will be improved [68-70]. CaCO_3 can be created by different ways, including precipitation, dry grinding, and wet grinding, and essential evaluations of CaCO_3 can be separated by size of particles, surface area and morphology [71-74].

In this research different amounts and percentages of the calcium carbonate (CaCO_3) and DIDP plasticizer added to the PVC blends to make samples, and studying the mechanical behavior for the samples. The samples made in Borsod-Chem at Miskolc-Hungary. The samples going through some tests and measurement. First of all, hardness mechanical test (shore A), then tensile strength test to know mechanical properties of new PVC including (elongation at break, tensile stress, tensile strain, and young modulus), electrical property by TSDC electrical test, FT-IR spectroscopy, then thermal characterization test by (DMA). After all measuring, the results will be comparing with the previously works of literatures. The aim of project is to investigate the new PVC composites with a better mechanical property for further application.

II. MATERIALS AND METHODS

2.1 Applied Materials

2.1.1 Type of: PVC S5070:

($\text{C}_2\text{H}_3\text{Cl}$)_n with a melting range between 140°C to 200°C. The glass transition temperature is between 75°C to 85 °C and flow temperature is 140°C.

2.1.2 Plasticizer: DIDP

Diisodecyl phthalate (DIDP: increase flexibility of plastic and workability.

2.1.3 Heat Stabilizer ADVASTAB TM181

Advastab TM181 is performances as a heat stabilizer.

2.1.4 WAX E

Wax E is a classified class of natural mixtures: softening.

2.1.5 Hydrocarb 95T CaCO_3

Micro sized calcium carbonate which is extremely fine, size between 0.7 -100 μm .

2.1.6 SOCAL 312 CaCO_3

Ultrafine calcium carbonate is near to nano size. The particle size is below 0.01 μm .

3.2 Preparation of Samples.
The detail of all PVC samples shown in Table.1

TABLE 1

PVC SAMPLES WITH DIFFERENT AMOUNTS OF FILLERS AND MIXING. CaCO_3 (1) IS MICRO SIZED PARTICLE OF CALCIUM CARBONATE, CaCO_3 (2) IS NANO PARTICLE SIZED OF CALCIUM CARBONATE

Material /Sgn	ST0	ST1-10	ST1-10U	ST2-10	ST2-10U	ST1-20	ST1-20U	ST2-20	ST2-20U
PVC S5070	100	100	100	100	100	100	100	100	100
DIDP	50	50	50	50	50	50	50	50	50
Advastab-TM181	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2
Wax E	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
CaCO_3 (1)		10	10UH			20	20UH		
CaCO_3 (2)				10	10UH			20	20UH

2.2 Characterizations and Experimental Part

2.2.1 Fourier Transformation Infrared Spectrometry (FTIR)

Fourier-Transformation-Infrared spectroscopy (FTIR) is a scientific system used to checking the surface of PVC, and the comprehend the design of individual particles and the structure of molecular combinations. The machine was used for testing the samples for the FT-IR test was BRUKER TENSOR27 in the Institute of Ceramic and Polymer Engineering. The software that was used in computer for reading and analyzing the measurement data from the machine is called OPUS. First of all, the plate cleaned by ethyl-alcohol (Ethanol, C_2H_5OH) to make waves more accurately, also the sample should be cleaned before the testing. Then putting the sample on the plate and fix it under 65 scans measurements and wait until the data and result will be import to the computer, and repeating the same procedure for all the samples.

2.2.2 Tensile Properties Test

Tensile strength test was carried out on testing machine INSTRON 5566 speed of 100 mm/min. and room temperature 20°C as shown in figure 6. All of samples were cut and divided to 3 parallel measurements with size (length: 60 mm, thickness:2 mm, and width:10 mm) for each sample as shown in figure 7. In tensile test four measurements will be study which is the stress, strain, elongation at break and young modulus.

2.2.3 Hardness Test- Shore (A)

The shore A hardness of the PVC samples was measured using a machine named by Zwick Roell (type: H04.3150) as shown in figure 8. The indenter was pressed with sufficient band force for 5 seconds into the plastic specimen vertically with room temperature 20°C, and repeated the same procedure for each sample 10 times, each time in different point on the surface of sample as shown in figure 9. The results recorded and the average of hardness and standard deviation will be calculating. The shore (A) hardness scale measures the hardness of flexible mold rubbers that scope in hardness from very soft and flexible, toward medium and rather flexible, and toward hard with almost no flexibility at all.

2.2.4 TSDC Electrical Properties Test

PVC samples will examine under electrical direct current. In terms of their electrical properties, specific volume resistivity (ρ) will be measure with standard (IEC 93) for each PVC sample. Direct current (DC) is electrical current which flows consistently in one direction. The current that flows in a flashlight or another appliance running on batteries is direct current. Selecting DC voltage on the multimeter will be (voltage $U_m=500V$) and electrode area ($A=44.15cm^2$). Thermal stimulated depolarization current (TSDC) is use to define the varying rules of charge distribution within insulation structure when it reaches the thermal equilibrium state and the specific parameters [112].

2.2.5 Thermal Characterization Test: Dynamic Mechanical Analysis (DMA)

Dynamic mechanical analysis (DMA) is a method for characterizing thermal behavior of PVC samples as a function of temperature, time, frequency, stress, atmosphere or an arrangement of these limits [74]. DMA measures stiffness and damping, these are reported as storage modulus and loss factor ($\tan \delta$) [26,40,43], and also describe the relaxation behavior of the PVC [4,6,22,44,53,69,72]. The DMA tests were measured by (Metravib 1Db DMA 25) machine. Tension and compression over a temperature range (-60 °C to 120°C) with heating rate of 2°C per minute at frequency 10Hz. The dimensions of PVC samples for DMA test are (length: 15 mm, width 12 mm, and thickness 1.2 mm).

III. RESULTS AND DISCUSSIONS

3.1 FT-IR Test Results and Discussion

FTIR procedures usually look at the PVC resin carbon- chlorine bond and plasticizer groups. The results of FT-IR spectras for all the samples was shown in Fig.1.

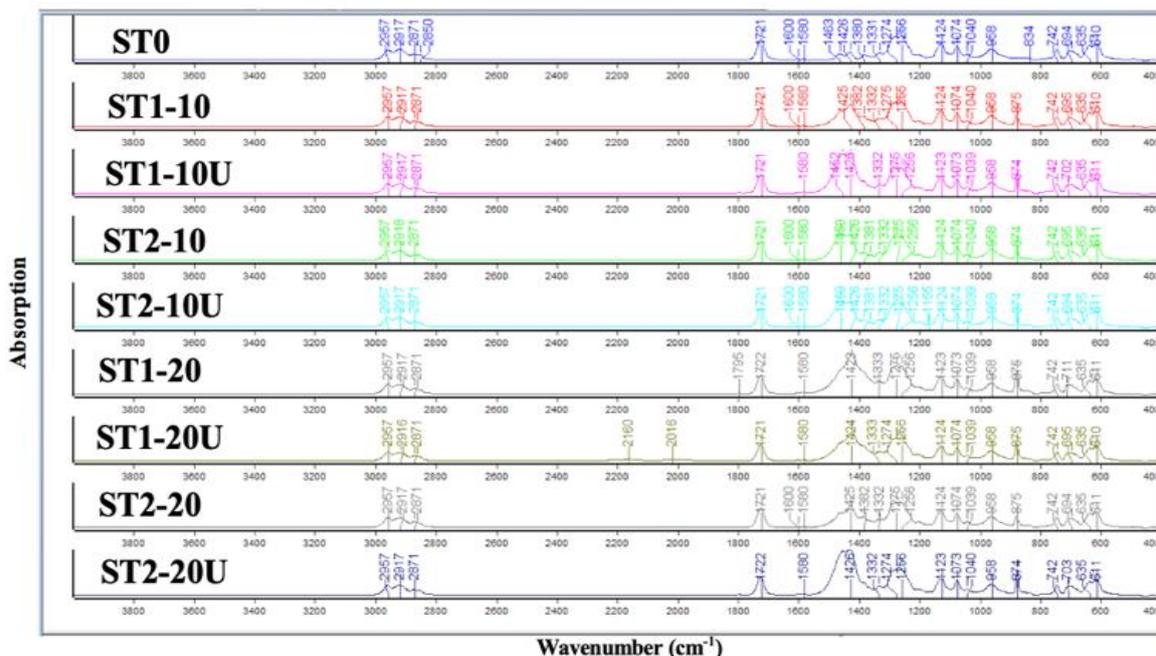


FIGURE 1: FT-IR spectras result for PVC samples.

FT-IR spectra of the plasticizers and the PVC samples were acquired. The main absorption bands in the infrared are shown in Table 2. Each sample can be observe the peaks of 2957 cm⁻¹ which is related to CH₃ stretching and at the peaks of 2916-1918 cm⁻¹ can be observe the CH₂ stretching which is a behavior of PVC polymer [80,84,101,102,104]. CH₂ angular deformation mode at 1331-1333 cm⁻¹ [101,102], and C-Cl stretching mode at 834-610 cm⁻¹ [84, 96, 101-104]. The difference between the pure PVC which is sample (ST0) and sample (ST2-20U) can be seen from the FT-IR spectra that the sample (ST2-20U) is more sensitive comparing to the pure PVC (ST0), and the ST2-20U sample is extra homogenous and the plasticizer on the surface is not obvious, but its inside sample everywhere and well mixed, that is make it the best one among all of the samples.

TABLE 2
MAIN INFRARED ABSORPTION BONDS OF THE PLASTICIZERS AND PVC

Wavenumber (cm ⁻¹)	Assignment	References
2957	Asymmetric Stretching CH ₃	84,101,102,104
2916-2918	Stretching CH ₂	101,102,104
2871	Symmetric stretching CH ₃	102
2850	CH ₂ Symmetric stretching	102,104
1795	C=O stretching	103,80
1721-1722	C=O stretching	102,80
1580-1600	C=C aromatic stretching	102
1423-1463	Deformation (Wagg), CH ₂ -Cl	101,104
1380-1382	CH ₃ Symmetric angular	102
1331-1333	CH ₂ Angular deformation	101,102
1255-1275	Cl-CH Out of plane angular deformation	102,104
1123-1124	C-H aromatic	102
1039-1074	Rocking, CH ₂	101,104
958	C-H or C-C aromatic	102
874-875	Stretching, C-Cl	84,103
834	Chain stretching	102,104
694-610	Stretching, C-Cl	96,101,102,104

3.2 Tensile Properties Test Results and Discussion

3.2.1 Tensile Stress and Strain Results

It can be ascertained from the figure 2 that the tensile strength is maximum at (13.71 MPa) in sample named (ST2-10U) which is contain 10 wt% of nano- CaCO₃ particle, by ultrasonic mixing, the results of tensile stress is shown in Fig.2.

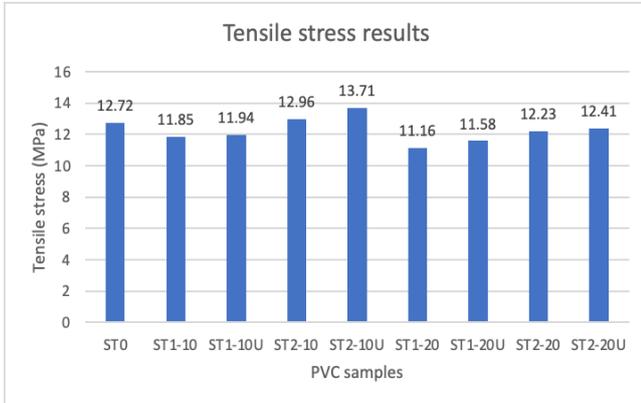


FIGURE 2. Tensile stress results

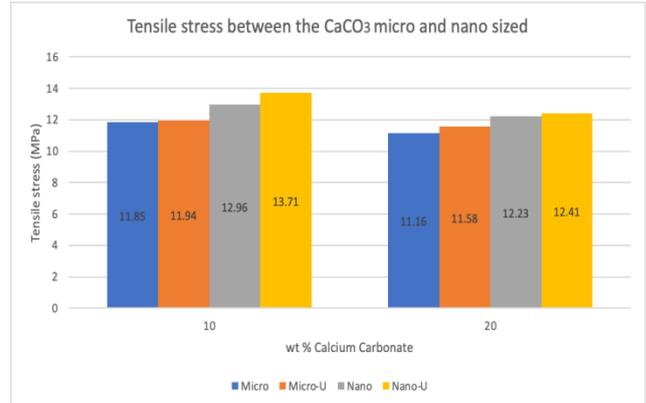


FIGURE 3. Comparing tensile strength between the CaCO₃ micro and nano sized particles

The reason behind increasing the tensile strength in the nano particle of CaCO₃ as shown in Fig.3 is that nano particles have excellent mechanical properties and unique properties that are not found in micro particle and nanocomposite products contain reinforcing or fillers in nanoscale. Most of the mechanical properties can be enhanced using nano scale particles this might be ascribed to the incredible similarity between nano CaCO₃ and PVC matrix.

The result of tensile strain is shown in Fig.4, the highest tensile strain is (428.33) which can be seen in two samples (ST2-10U and ST2-20U).

The results showed that the tensile strain of nano particle of CaCO₃ is higher than micro sized particle of CaCO₃.

Nano particle of calcium carbonate affected and increasing in tensile strain of the PVC more than the micro particles. The results of growth could be recognized to the huge boundary area between nano-sized particles and matrix.

3.2.2 Elongation at Break Results and Discussion

The maximum elongation is (6.97) which can be seen in sample (ST2-10U) which contain 10 wt % nano particles of CaCO₃. Elongation at break initially increases and then decreases quickly when the quantity of CaCO₃ amount is greater than 10 wt % as shown in Fig.5. Comparing the PVC samples contain micro size of CaCO₃ particles to pure PVC, the pure PVC has higher elongation which increases in homogeneity and reduces the elongation

The nano particle affects more in raising the elongation at break of the PVC sample PVC samples filled nano particle of CaCO₃ have higher elongation than micro.

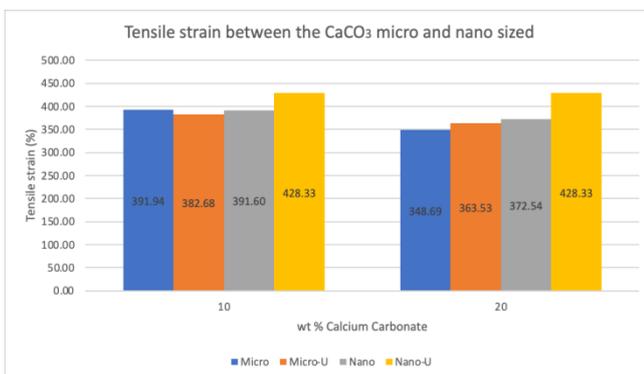


FIGURE 4. Tensile strain results

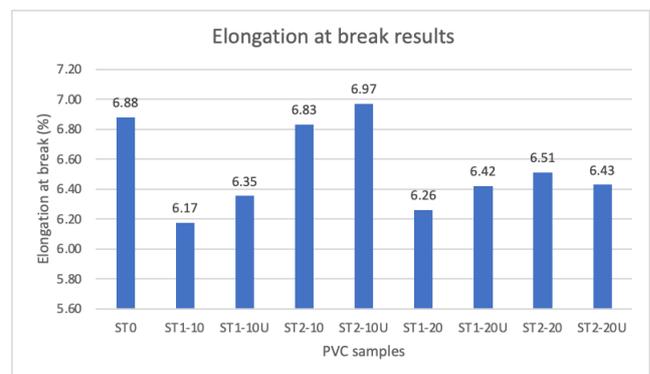


FIGURE 5. Elongation at break results

3.2.3 Young Modulus Results and Discussion

The results showed that young modulus increase by increasing the amount of CaCO_3 as shown in Fig.6 and it reached the highest value in 10 wt % of CaCO_3 , but above 10 wt % young modulus gradually decreasing. Comparing the micro to nano CaCO_3 particle in PVC samples, the results showed that using nano sized particles of CaCO_3 in PVC sample have more positive affect than micro sized CaCO_3 particles.

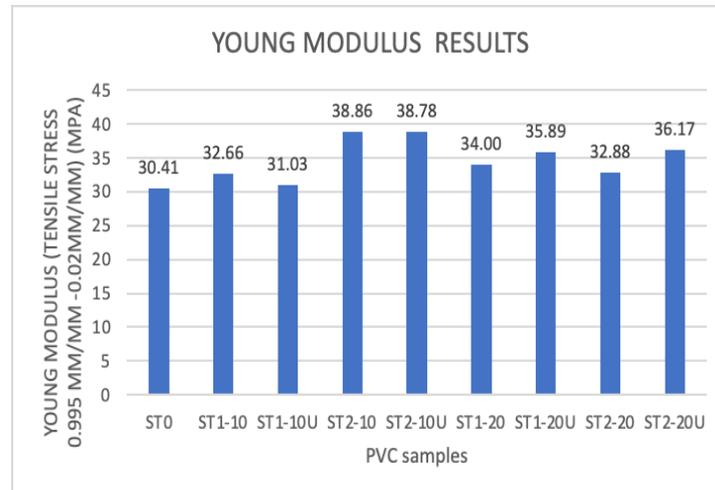


FIGURE 6: Young modulus Results

3.3 Hardness (Shore A) Results and Discussion

The highest value is (93.52 °Sh) for sample (ST2-10U) which contain 10 wt % nano particles of CaCO_3 . The results showed in Fig.7. PVC samples have great resistance to indentation and they are hard materials. The raise in hardness is more considerable in nano CaCO_3 filled PVC compared to that of micro CaCO_3 . The way of mixing also affected the raise of hardness, obviously the PVC samples with ultrasonic mixing results are much higher than the normal mixing of PVC sample.

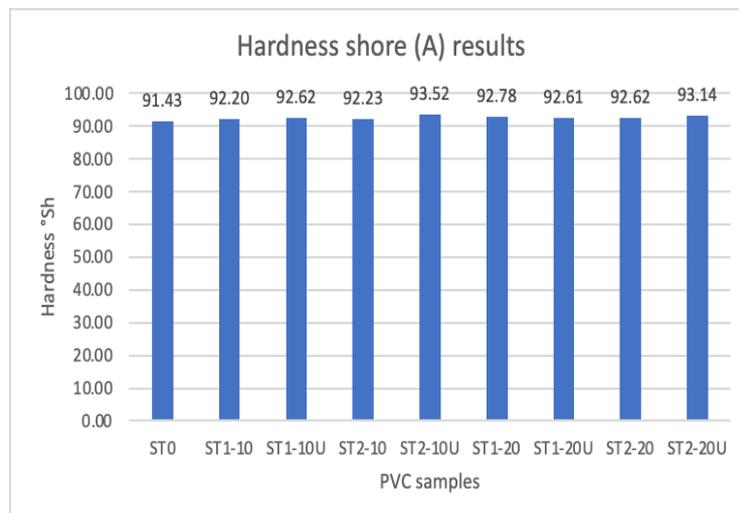


FIGURE 7: Hardness (shore A) results

3.4 TSDC Electrical Test Results and Discussion

The smallest electrical resistance: (ST1-10U) which is (0.9 M Ω), highest electrical resistance: (ST2-20U) which is (6.1 M Ω). The maximum volume resistivity : (ST1-10U) with value (3.34×10^{12} Ωcm) which is a PVC refilled with micro particles of CaCO_3 ultrasonic mixed as showed in Fig. 8 and Fig. 9. The specific volume resistivity in PVC micro sized CaCO_3 normal mixing is reducing by increasing the amount of CaCO_3 to 10 wt %, and slowly increase in 20 wt%. The specific volume resistivity in PVC micro sized CaCO_3 ultrasonic mixing is rapidly growth by increasing the amount of CaCO_3 to 10 wt %, and slowly decrease in 20 wt%. The PVC with nano CaCO_3 particles showed improvement in insulation resistance when compared to unmodified PVC.

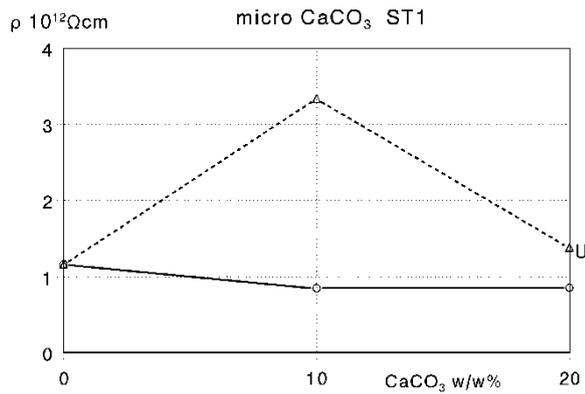


FIGURE 8. Result of electrical resistance and specific volume resistivity of micro sized CaCO₃ filled in PVC

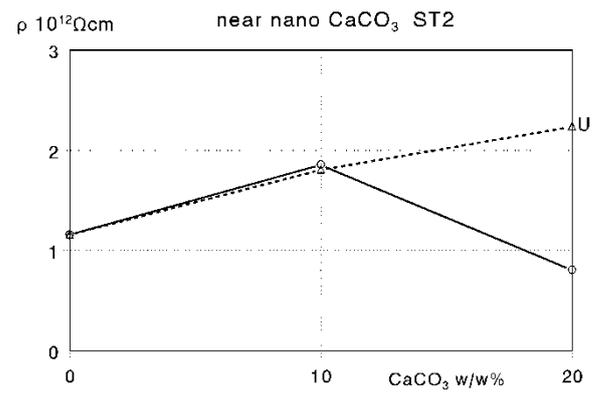


FIGURE 9. Result of electrical resistance and specific volume resistivity of nano sized CaCO₃ filled in PVC

IV. RESULTS AND DISCUSSION

4.1 Thermal Characterization Test (DMA)

The storage modulus of PVC decreased as a function of temperature that indicated that samples became in the rubber mood by increasing temperature. The storage modulus can be increased concurrently by the presence of CaCO₃ as showed in Fig. 10 and Fig.11.

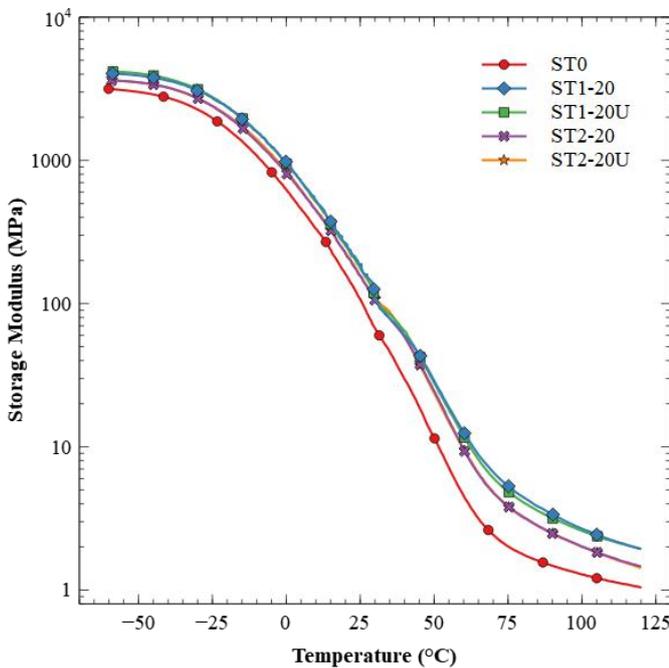


FIGURE 10. Dynamic storage modulus results for PVC samples

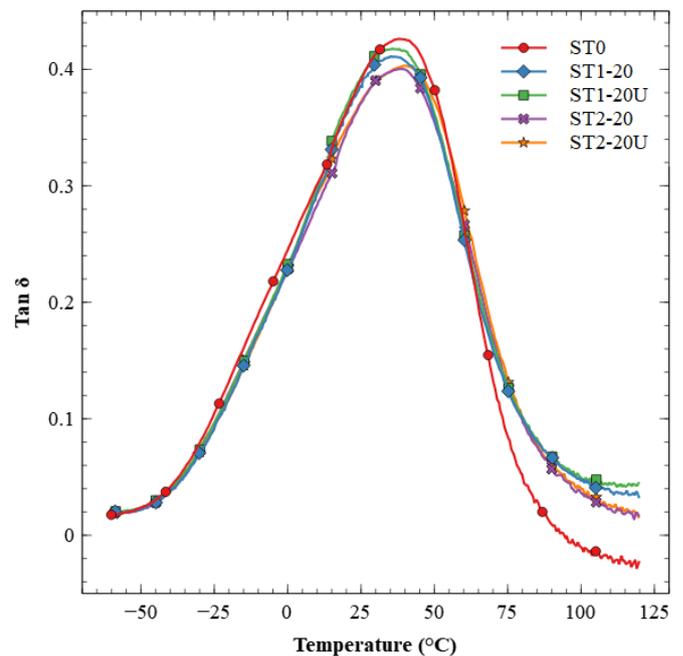


FIGURE 11. Loss factor (tan δ) results for PVC samples

The storage modulus of PVC decreased as a function of temperature that indicated that samples became in the rubber mood by increasing temperature [69]. The pure PVC (ST0) has storage modulus (3164.18 MPa) at temperature (-60 °C) which is lowest storage modulus comparing to other PVC samples at temperature (-60 °C). The highest storage modulus is in samples (ST1-20U) which it has (4180.93 MPa) at temperature (-60 °C) and (1.95 MPa) at temperature (120 °C). The storage modulus can be increased concurrently by the presence of CaCO₃ [78,91]. Loss factor (tan δ) of PVC samples is shown in Table 2. The glass transition of pure PVC is 37.9 °C and it is higher than comparing to the PVC micro- CaCO₃, but lower than nano-CaCO₃ samples.

TABLE 3
GLASS TRANSITION TEMPERATURE OF PVC SAMPLES

PVC samples	Glass transition temperature (T_g) ($^{\circ}\text{C}$)
ST0	37.9
ST1-20	35.2
ST1-20U	35.5
ST2-20	38.7
ST2-20U	39.7

Comparing the glass transition temperature between micro- CaCO_3 and nano- CaCO_3 samples, it is obviously increased in nano- CaCO_3 [91,95,98,100], and the way of mixing affected the glass transition because the samples which mixed by ultrasonic has higher glass transition than those samples which mixed by normal mixing. It can be found that the stability properties of nano- CaCO_3 are better than that of the pure PVC resin. [97]. The DMA results are shown that loss factor ($\tan \delta$) of PVC/nano- CaCO_3 composites were higher than those of PVC, and all these indicate that the interfacial interaction between PVC and nano- CaCO_3 particles can be improved by modification of nano- CaCO_3 particles surfaces [98].

V. SUMMARY

PVC samples prepared for this research with micro and nano size of calcium carbonate particle with different phr (0 wt%, 10 wt% and 20 wt%), and different type of mixing. DIDP used as plasticizer added to PVC samples by 50 phr, which applied in the creation of plastic and covering to improve flexibility of plastic, and (Advastab TM181) added to PVC samples by 1.2 phr, which used for gives generally excellent long-haul tones and colors, and impact strength maintenance, and (wax E) added to PVC samples by 0.4 phr for softening to give low consistency of PVC. Roll-milling mixes were obtained utilizing a blender at 165°C for 5 minutes in a rotational speed. The obtained samples were shaped as 2-mm-thick sheets, and different ratio of each plasticizer and two type of mixing which is ultrasonic mixing and normal mixing. The ultrasonic electronic generator switches AC line power to a 20 kHz signal that powers a piezoelectric convertor/transducer.

The PVC samples were examined by FT-IR spectra considering appearance of the PVC resin carbon- chlorine bond and plasticizer groups of the plasticizers to study about the surface of the samples. The main absorption bands in the infrared were acquired. All samples observed the peaks of 2957 cm^{-1} which is related to CH_3 stretching and at the peaks of 2916-1918 cm^{-1} can be observe the CH_2 stretching which is a behavior of PVC polymer, and CH_2 Angular deformation mode at 1331-1333 cm^{-1} . Also C-Cl stretching mode at 834-610 cm^{-1} . The sample (ST2-20U) which contained 20 wt % nano particles of CaCO_3 with ultrasonic mixing acquired that it is extra homogenous and the plasticizer on the surface is not obvious, but its inside sample everywhere and well mixed, that is make it the best one among all of the samples.

The PVC samples were examined by tensile stress which measured the strength of a material, in this manner, it alludes to a power that endeavors to pull separated or stretch a material. The tensile strength measured highest in the sample (ST2-10U) which is 10 wt % of nano particle of CaCO_3 with ultrasonic mixing, and greater than any other compositions with value of (13.71 MPa). By increasing the amount of CaCO_3 above 10 % to 20 %, tensile stress decreased. The highest tensile strain is (428.33) which can be seen in two samples (ST2-10U and ST2-20U) which both of them contain nano particles of CaCO_3 ultra sonic mixed with 10 wt % and 20 wt %. The results showed that by increasing the amount of nano- CaCO_3 from 10 % to 20 %, does not affect the strain of the PVC and stay constant. Nano particle of calcium carbonate affected and increasing in tensile strain of the PVC more than the micro particles. The results of growth could be recognized to the huge boundary area between nano-sized particles and matrix.

The results showed that the tensile strain of nano particle of CaCO_3 is higher than micro sized particle of CaCO_3 .

The maximum elongation was (6.97) which can be seen in sample (ST2-10U) which contain 10 wt % nano particles of CaCO_3 , elongation at break initially increases until when the amount of CaCO_3 reach 10 wt %, and then decreases rapidly when the amount of CaCO_3 is greater than 10 wt %. Comparing the PVC samples contain micro size of CaCO_3 particles to pure PVC, the pure PVC has higher Elongation. The result showed that PVC samples filled nano particle of CaCO_3 have higher elongation than micro size of CaCO_3 .

Young modulus examined and the results showed that young modulus increase by increasing the amount of CaCO_3 and it reached the highest value in 10 wt % of CaCO_3 , but above 10 wt % Young modulus gradually decreasing. The results showed that using nano sized particles of CaCO_3 in PVC sample have more positive affect than micro sized CaCO_3 particles.

Hardness test examined and according to the results, the highest value in sample (ST2-10U) which is (93.52 °Sh) which contain 10 wt % nano particles of CaCO_3 , and the lowest value is (91.43°Sh) for sample (ST0) which is pure PVC without CaCO_3 particles. The results showed that PVC samples have great resistance to indentation and they are hard materials. The raise in hardness is more considerable in nano CaCO_3 filled PVC compared to that of micro CaCO_3 . The results showed that CaCO_3 affected the hardness of PVC and raise it respectively. The way of mixing also affected the raise of hardness, obviously the PVC samples with ultrasonic mixing results are much higher than the normal mixing of PVC sample.

The lowest value of standard deviation can be seen in sample (ST0) which is 0.11, and the highest value of standard deviation can be seen in sample (ST1-10) which is 0.45 as shown in figure 26. After that the standard deviation reduced in sample (ST1-10U) to 0.27. Then the value growth in sample (ST2-10) which is 0.31. The standard deviation decreases in sample (ST2-10U) to 0.23, then increase respectively in sample (ST1-20) to 0.24 and rise again sample (ST1-20U) to 0.29. After that it decreased in sample (ST2-20) to 0.19. Finally, in the last sample (ST2-20U) it increased to 0.28.

PVC samples examined with TSDC, electrical properties of them detected. In terms of their electrical properties, specific volume resistivity (ρ) with standard (IEC 93).The sample (ST1-10U) has the smallest electrical resistance which is (0.9 M Ω) and sample (ST2-20U) has the largest electrical resistance which is (6.1 M Ω). The PVC with nano CaCO_3 particles showed improvement in insulation resistance when compared to unmodified PVC. The maximum volume resistivity can be seen in sample (ST1-10U) with value ($3.34 \times 10^{12} \Omega\text{cm}$) which is a PVC refilled with micro particles of CaCO_3 Ultrasonic mixed. The specific volume resistivity in PVC micro sized CaCO_3 normal mixing is reducing by increasing the amount of CaCO_3 to 10 wt %, and slowly increase in 20 wt%. The specific volume resistivity in PVC micro sized CaCO_3 ultrasonic mixing is rapidly growth by increasing the amount of CaCO_3 to 10 wt %, and slowly decrease in 20 wt%. In contrast, the specific volume resistivity in PVC nano sized CaCO_3 normal mixing is growth by increasing the amount of CaCO_3 to 10 wt %, and rapidly decrease in 20 wt%. The specific volume resistivity in PVC nano sized CaCO_3 ultrasonic mixing is rapidly growth by increasing the amount of CaCO_3 to 10 wt %, and continue rising in 20 wt%.

Thermal characterization of PVC samples examined by DMA. Storage modulus and loss factor ($\tan \delta$) of PVC decreased as a function of temperature, that indicated that samples became in the rubber mood by increasing temperature. The storage modulus can be increased concurrently by the presence of CaCO_3 . The glass transition of pure PVC is higher than PVC micro- CaCO_3 , but lower than PVC nano- CaCO_3 samples. The way of mixing affected the glass transition because the samples which mixed by ultrasonic has higher glass transition than those samples which mixed by normal mixing. It can be found that the stability properties of nano- CaCO_3 are better than that of the pure PVC resin. Future work will be checking the thermal properties such as heat stability and flame retardancy, and colorization of the PVC samples.

In the PVC compounding industry the fillers are usually added in the high speed mixer. The general problem is the agglomeration of the filler particles. The lower is the particle size the higher is the danger of agglomeration. Therefore the advantages of the low particle size cannot be utilized. Nowadays the ultrasonic mixing is easily accessible even in industrial size therefore I tried to compare the mixing methods.

The mixing ratio is a question of application; it is decided at creating basic formulation. I have found that the ultrasonic mixing produces considerably better dispersion, by these way better properties. The limit of ultrasonic mixing is the filler/fluid ratio. The electrical industry, namely cable insulating materials, is the industrial sector where ultrasonic mixing is advantageous.

REFERENCES

- [1] K.A. Carrado (2000): *Synthetic organo- and polymer-clays: Preparation, characterization, and materials applications*, **Applied Clay Science**, vol. 17(1-2), pp. 1-23. DOI:10.1016/s0169-1317(00)00005-3
- [2] S.L. Rosen, J.Wiley & Sons (1993): *Fundamental Principles of Polymeric Materials*, **Applications of electroactive polymers**, 2nd edition, pp. 343-344. DOI:10.1002/pi.1994.210330324
- [3] K. Endo (2002). *Synthesis and structure of poly(vinyl chloride)*, **Progress in Polymer Science**, vol. 27(10), pp.2021-2054. DOI:10.1016/S0079-6700(02)00066-7
- [4] Cui, J., Cai, Y., Yuan, W., Lv, Z., Zhang, C., & Xu, S. (2015). *Preparation of PMMA grafted calcium carbonate whiskers and its reinforcement effect in PVC*, **Polymer Composites**, vol. 38(12), pp.2753-2761. DOI:10.1002/pc.23873

- [5] Xie, X., Liu, Q., Li, R. K., Zhou, X., Zhang, Q., Yu, Z., & Mai, Y. (2004): *Rheological and mechanical properties of Poly (Vinyl Chloride)/CaCO₃ nanocomposites prepared by in situ polymerization*, **Polymer**, vol. 45(19), pp. 6665-6673.
DOI: 10.1016/j.ijpvp.2011.01.002
- [6] Laiarinandrasana L, Gaudichet E, Oberti S, Devilliers C (2011). *Effects of aging on the creep behaviour and residual lifetime assessment of polyvinyl chloride (PVC) pipes*, **International Journal of Pressure Vessels**, vol. 88(2-3), pp. 99-108.
DOI: 10.1016/j.polymer.2004.07.045
- [7] Li B. (2003). *Influence of polymer additives on thermal decomposition and smoke emission of poly (vinyl chloride)*. **Polymer Degradation and Stability**, vol. 82(3), pp.467-476. DOI:10.1016/S0141-3910(03)00201-5
- [8] Krongauz VV, Lee YP, Bourassa A. (2011). *Kinetics of thermal degradation of poly (vinyl chloride)*, **Journal of Thermal Analysis and Calorimetry**, vol. 106(1), pp.139-149. DOI:10.1007/s10973-011-1703-6
- [9] E. Yousif, A. Ahmed, R. Abood, N. Jaber, R. Noaman & Rahimi Yusop (2015): *Poly(vinyl chloride) derivatives as stabilizers against photodegradation*, **Journal of Taibah University for Science**, vol. 9:2, pp. 203-212. DOI: [10.1016/j.jtusci.2014.10.003](https://doi.org/10.1016/j.jtusci.2014.10.003)
- [10] Amar, Z., Chabira, S., Sebaa, M., & Ahmed, B. (2019). *Structural Changes Undergone During Thermal Aging and/or Processing of Unstabilized, Dry-blend and Rigid PVC, Investigated by FTIR-ATR and Curve Fitting*, **Annales De Chimie - Science Des Matériaux**, vol. 43(1), pp.59-68. DOI:10.18280/acsm.430109
- [11] Marcilla, A., García, S., García-Quesada, J. (2004): *PVC plasticizers receive top marks for migration. Study of the Migration of PVC Plasticizers*, **Journal of Analytical and Applied Pyrolysis**, vol. 71(2), pp. 457-463. DOI:10.1016/s1464-391x(04)00292-2
- [12] Ghanbar, S., Yousefzade, O., Hemmati, F., & Garmabi, H. (2014). *Microstructure and thermal stability of polypropylene/bagasse composite foams: Design of optimum void fraction using response surface methodology*, **Journal of Thermoplastic Composite Materials**, vol. 29(6), pp.799-816. DOI:10.1177/0892705714535795
- [13] Ma, J., Duan, Z., Xue, C., & Deng, F. (2013): *Morphology and mechanical properties of EVA/OMMT nanocomposite foams*, **Journal of Thermoplastic Composite Materials**, vol. 26(4), pp.555-569. DOI:10.1177/0892705712458943
- [14] Bose, S., & Mahanwar, P. (2004): *Effect Of Flyash On The Mechanical, Thermal, Dielectric,Rheological And Morphological Properties Of Filled Nylon 6*, **Journal of Minerals and Materials Characterization and Engineering**, vol. 03(02), pp.65-72.
DOI:10.4236/jmmce.2004.32007
- [15] Khoshnoud, P., & Abu-Zahra, N. (2016): *Properties of rigid polyvinyl chloride foam composites reinforced with different shape fillers*, **Journal of Thermoplastic Composite Materials**, vol. 30(11), pp.1541-1559. DOI:10.1177/0892705716646417
- [16] Han, R., Nie, M. & Wang, Q. (2017): *Continuously enhanced hoop strength of rotation-extruded polypropylene pipe via self-assembly β nucleating agent with different aspect ratio*, **Journal of Polymer Research**, vol. 204, pp.1-10
DOI:10.1007/s10965-017-1379-0
- [17] DOI:10.1007/s10965-017-1379-0
- [18] Jemii, H., Bahri, A., Boubakri, A. et al. (2020): *On the mechanical behaviour of industrial PVC pipes under pressure loading: experimental and numerical studies*, **Journal of Polymer Research**, vol. 27 (240), pp.1-13. DOI:10.1007/s10965-020-02222-1
- [19] Demir, H., Sipahioğlu, M., Balköse, D., & Ülkü, S. (2008): *Effect of additives on flexible PVC foam formation*, **Journal of Materials Processing Technology**, vol. 195(1-3), pp.144-153. DOI:10.1016/j.jmatprotec.2007.04.123
- [20] Djidjelli H, Sadoun T, Benachour D (2000): *Effect of plasticizer nature and content on the PVC stability and dielectric properties*, **Journal of Applied Polymer Science**, vol. 78, pp.685-691.
DOI: 10.1002/1097-4628(20001017)78:3<685::AID-APP250>3.0.CO;2-F
- [21] Mendizabal, E., Cruz, L., Jasso, C., Burillo, G., & Dakin, V. (1996): *Radiation crosslinking of highly plasticized PVC*, **Radiation Physics and Chemistry**, vol. 47(2), pp. 305-309. DOI:10.1016/0969-806x(94)00183-k
- [22] Ueno K, Uda I, Tada S (1991) *Radiation-crosslinked polyethylene for wire and cable applications*. **Int International Journal of Radiation Applications and Instrumentation. Part C. Radiation Physics and Chemistry**, vol. 37, pp. 89-91.
DOI: 10.1016/1359-0197(91)90203-E
- [23] Price D, Pyrah K, Hull TR, Milnes GJ, Ebdon JR, Hunt BJ, Joseph P (2002): *Flame retardance of poly (methyl methacrylate) modified with phosphorus-containing compounds*, **journal of polymer degradation and stability**, vol. 77, pp. 227-233.
DOI:10.1016/S0141-3910(02)00038-1
- [24] Basfar AA (2002): *Flame retardancy of radiation cross-linked poly(vinyl chloride) (PVC) used as an insulating material for wire and cable*, **journal of polymer degradation and stability**. 77, pp. 221-226. DOI:10.1016/S0141-3910(02)00037-X
- [25] Palin, L.; Rombolà, G.; Milanesio, M.; Boccaleri, E (2019): *The Use of POSS-Based Nanoadditives for Cable-Grade PVC: Effects on Its Thermal Stability*, **Polymers**, vol. 11(7), pp. 1-15. DOI: 10.3390/polym11071105
- [26] Aljoumaa, K., & Aji, Z. (2016): *Thermal and mechanical properties of irradiated poly(vinyl chloride)/calcium carbonate composite*, **Journal of Radioanalytical and Nuclear Chemistry**, vol. 311(1), pp.15-22. DOI:10.1155/2017/2717848
- [27] Touaiti, F., Pahlevan, M., Nilsson, R., Alam, P., Toivakka, M., Ansell, M., & Wilen, C. (2013): *Impact of functionalised dispersing agents on the mechanical and viscoelastic properties of pigment coating*. **Progress in Organic Coatings**, vol. 76(1), pp.101-106.
DOI:10.1016/j.porgcoat.2012.08.013
- [28] Bahari, S. A., Grigsby, W. J., & Krause, A. (2017): *Flexural Properties of PVC/Bamboo Composites under Static and Dynamic-Thermal Conditions: Effects of Composition and Water Absorption*. **International Journal of Polymer Science**, vol. 2017 pp.1-8.
DOI:10.1007/s10967-016-4873-7

- [29] Etienne, S., Becker, C., Ruch, D., Germain, A., & Calberg, C. (2009): *Synergetic effect of poly(vinyl butyral) and calcium carbonate on thermal stability of poly(vinyl chloride) nanocomposites investigated by TG-FTR-MS*, **Journal of Thermal Analysis and Calorimetry**, vol. 100(2), pp.667-677. DOI:10.1007/s10973-009-0443-3
- [30] K. P. Schlickmann, J. L. L. Howarth, D. A.Kasper, A. P. T. Testa. (2019): *Effect of The Incorporation of Micro and Nanoparticles of Calcium Carbonate in Poly (Vinyl Chloride) Matrix for Industrial Application*. **Materials Research**, vol. 22, ISSN: 2018-0870, DOI:10.1590/1980-5373-mr-2018-0870
- [31] Abdulla, N. A. (2017): *Concrete filled PVC tube: A review*, **Construction and Building Materials**, vol. 156, pp.321-323. DOI: 10.1016/j.conbuildmat.2017.08.156
- [32] Shahamirifard, S. A., & Ghaedi, M. (2017): *Design and construction of a new optical solid-state mercury(ii) sensor based on PVC membrane sensitized with colloidal carbon dots*, **New Journal of Chemistry**, vol. 41(20), pp.11533-11535. DOI:10.1039/c7nj02421e
- [33] Mazitova, A., Aminova, G., Maskova, A., Yagafarova, G., & Mazitov, R. (2017): *New plasticizers for PVC-compositions in construction*. **Nanotechnologies in Construction: A Scientific Internet-Journal**, vol. 9(4), pp.48-50. DOI:10.15828/2075-8545-2017-9-4-48-63
- [34] Wang, S. P., Chen, W. J., Wang, Z. H., Gong, X. Z., Zhao, C. Z., & Ma, L. P. (2014): *Life Cycle Assessment of PVC-U Plastic Window Production*, **Key Engineering Materials**, vol. 599, pp. 319–323. DOI:10.4028/www.scientific.net/kem.599.319
- [35] Luciani, V., Bonifazi, G., Rem, P., & Serranti, S. (2015): *Upgrading of PVC rich wastes by magnetic density separation and hyperspectral imaging quality control*, **Waste Management**, vol. 45, pp.118-125. DOI:10.1016/j.wasman.2014.10.015
- [36] V. Luciani, S. Serranti, G. Bonifazi, F. D. Maio & Peter Rem (2013): *Quality control in the recycling stream of PVC from window frames by hyperspectral imaging*, **Optical Sensors**, vol. 8774 pp. 1-3. DOI:10.1117/12.2014755
- [37] Facca, A. G., Kortschot, M. T., & Yan, N. (2007): *Predicting the tensile strength of natural fibre reinforced thermoplastics*, **Composites Science and Technology**, 67(11-12), pp.2454-2466. DOI:10.1016/j.compscitech.2006.12.018.
- [38] Ali, S. F., Batouti, M. E., Abdelhamed, M., & Rafeq, E. E. (2020): *Formulation and characterization of new ternary stable composites: Polyvinyl chloride-wood flour- calcium carbonate of promising physicochemical properties*, **Journal of Materials Research and Technology**, vol. 9(6), pp.12840-12854. DOI:10.1016/j.jmrt.2020.08.113
- [39] Ciacci, L., Passarini, F., & Vassura, I. (2017): *The European PVC cycle: In-use stock and flows*. **Resources, Conservation and Recycling**, vol. 123, pp.108-116. DOI:10.1016/j.resconrec.2016.08.008
- [40] Everard, M., & Blume, R. (2019): *Additive sustainability footprint: Rationale and pilot evaluation of a tool for assessing the sustainable use of PVC additives*, **Journal of Vinyl and Additive Technology**, vol. 26(2), pp.196-208. DOI:10.1002/vnl.21733
- [41] C.B. Patil, U.R. Kapadi, D. G. Hundiware, P.P Mahulikar (2009): *Preparation and characterization of poly (vinyl chloride) calcium carbonate nanocomposites via melt intercalation*, **Journal of Materials Science**, vol. 44(12), pp. 3118- 3124. DOI: 10.1007/s10853-009-3414-y
- [42] Fugit, J., Taverdet, J., Gauvrit, J., & Lanteri, P. (2003): *Treatment of plasticized PVC to reduce plasticizer/solvent migration: Optimization with an experimental design*, **Polymer International**, vol. 52(5), pp. 670-675. DOI:10.1002/pi.971
- [43] Karayildirim, T., Yanik, J., Yuksel, M., Saglam, M., Vasile, C., & Bockhorn, H. (2006): *The effect of some fillers on PVC degradation*, **Journal of Analytical and Applied Pyrolysis**, vol. 75(2), pp.112-119. DOI:10.1016/j.jaap.2005.04.012
- [44] Garcia, D., Balart, R., Parres, F., & López, J. (2007): *Characterization of blends of poly(vinyl chloride) waste for building applications*. **Journal of Materials Science**, vol. 42(24), pp.10143-10151. DOI:10.1007/s10853-007-2067-y
- [45] Chiellini, F., Ferri, M., Morelli, A., Dipaola, L., & Latini, G. (2013): *Perspectives on alternatives to phthalate plasticized poly(vinyl chloride) in medical devices applications*, **Progress in Polymer Science**, vol. 38(7), pp.1067-1088. DOI:10.1016/j.progpolymsci.2013.03.001
- [46] Fang, Y., Wang, Q., Guo, C., Song, Y., & Cooper, P. A. (2013): *Effect of zinc borate and wood flour on thermal degradation and fire retardancy of Polyvinyl chloride (PVC) composites*, **Journal of Analytical and Applied Pyrolysis**, vol. 100, pp. 230-236. DOI:10.1016/j.jaap.2012.12.028
- [47] Kun, E., & Marossy, K. (2012): *Evaluation Methods of Antimicrobial Activity of Plastics*, **Materials Science Forum**, vol. 729, pp.430-435. DOI:10.4028/www.scientific.net/msf.729.430
- [48] Pinguet, J., Kerckhove, N., Eljezi, T., Lambert, C., Moreau, E., Bernard, L., Richard, D. (2019): *New SPE-LC-MS/MS method for the simultaneous determination in urine of 22 metabolites of DEHP and alternative plasticizers from PVC medical devices*, **Talanta**, vol. 198, pp. 377-389. DOI:10.1016/j.talanta.2019.01.115
- [49] Grossu, E., Opran, C. G., & Ditu, L. M. (2019): *Polyvinyl Chloride Antibacterial Recipes for Medical Devices*, **Materials Science Forum**, vol. 957, pp. 409-416. DOI:10.4028/www.scientific.net/msf.957.409
- [50] Saujanya, C., Ashamol, Padalkar, S., & Radhakrishnan, S. (2001): *Control of nanoparticle size of fillers by polymer blend technique*, **Journal of Polymer**, vol. 42(5), pp.2255-2258. DOI:10.1016/s0032-3861(00)00474-2
- [51] Pan, M., Shi, X., Li, X., Hu, H., & Zhang, L. (2004): *Morphology and properties of PVC/clay nanocomposites via in situ emulsion polymerization*, **Journal of Applied Polymer Science**, vol. 94(1), 277-286. DOI:10.1002/app.20896
- [52] Ren, T., Yang, J., Huang, Y., Ren, J., & Liu, Y. (2005): *Preparation, characterization, and properties of poly (vinyl chloride)/organophilic-montmorillonite nanocomposites*, **Polymer Composites**, vol. 27(1), 55-64. DOI:10.1002/pc.20161

- [53] Haiyan, H., Mingwang, P., Xiucuo, L., Xudong, S., & Liucheng, Z. (2004): *Preparation and characterization of poly (vinyl chloride)/organoclay nanocomposites by in situ intercalation*, **Polymer International**, vol. 53(2), 225-231. DOI:10.1002/pi.1309
- [54] Peprnicek, T., Duchet, J., Kovarova, L., Malac, J., Gerard, J., & Simonik, J. (2006): *Poly(vinyl chloride)/clay nanocomposites: X-ray diffraction, thermal and rheological behaviour*, **Polymer Degradation and Stability**, vol. 91(8), pp. 1855-1860. DOI:10.1016/j.polymdegradstab.2005.11.003
- [55] Yalcin, B., & Cakmak, M. (2004): *The role of plasticizer on the exfoliation and dispersion and fracture behavior of clay particles in PVC matrix: A comprehensive morphological study*, **Polymer**, vol. (19), pp. 6623-6638. DOI:10.1016/j.polymer.2004.06.061
- [56] Asif, K. M., Sarwar, M. I., Rafiq, S., & Ahmad, Z. (1998): *Properties of PVC-titania hybrid materials prepared by the sol-gel process*, **Polymer Bulletin**, vol. 40(4-5), pp. 583-590. DOI:10.1007/s002890050294
- [57] Chen, G. (2007): *Preparation of a poly (vinyl chloride)/layered double hydroxide nanocomposite with a reduced heavy-metal thermal stabilizer*, **Journal of Applied Polymer Science**, vol. 106 (2), pp.817-820. DOI:10.1002/app.26514
- [58] Pan, M.W.; Shi, X.D.; Li, X.C.; Hu, H.Y.; Zhang, L.C. (2004): *Morphology and properties of PVC/clay nanocomposites via in situ emulsion polymerization*, **Journal of Applied Polymer Science**, vol. 94, pp. 277. DOI: doi.org/10.1002/app.20896
- [59] Krzewki, R. J., & Collins, E. A. (2006): *Rheology of PVC compounds. I. Effect of processing variables on fusion*, **Journal of Macromolecular Science, Part B**, vol. 20(4), pp. 443-464. DOI:10.1080/00222348108205069
- [60] Tao, Y., Mao, Z., Yang, Z., & Zhang, J. (2020): *CaCO₃ as a new member of high solar-reflective filler on the cooling property in polymer composites*, **Journal of Vinyl and Additive Technology**, vol.26 pp. 1-12. DOI:10.1002/vnl.21801
- [61] Wu, D., Wang, X., Song, Y., & Jin, R. (2004): *Nanocomposites of poly (vinyl chloride) and nanometric calcium carbonate particles: Effects of chlorinated polyethylene on mechanical properties, morphology, and rheology*, **Journal of Applied Polymer Science**, vol. 92(4), pp. 2714-2723. DOI: 10.1002/app.20295
- [62] Liu, J., Pancera, S., Boyko, V., Gummel, J., Nayuk, R., & Huber, K. (2012): *Impact of Sodium Polyacrylate on the Amorphous Calcium Carbonate Formation from Supersaturated Solution*, **Langmuir**, vol. 28(7), pp.3593-3605. DOI:10.1021/la203895d
- [63] Wallace, A. F., Hedges, L. O., Fernandez-Martinez, A., Raiteri, P., Gale, J. D., Waychunas, G. A., Yoreo, J. J. (2013): *Microscopic Evidence for Liquid-Liquid Separation in Supersaturated CaCO₃ Solutions*, **Science**, vol. 341 (6148), pp. 885-889. DOI:10.1126/science.1230915
- [64] Fekete, E., Pukánszky, B., Tóth, A., & Bertóti, I. (1990): *Surface modification and characterization of particulate mineral fillers*. **Journal of Colloid and Interface Science**, vol. 135(1), pp.200-205. DOI:10.1016/0021-9797(90)90300-d
- [65] Chen, X., Zhu, Y., Guo, Y., Zhou, B., Zhao, X., Du, Y., Wang, Z. (2010): *Carbonization synthesis of hydrophobic CaCO₃ at room temperature*, **Colloids and Surfaces A: Physicochemical and Engineering Aspects**, vol. 353(2-3), pp. 97-105. DOI:10.1016/j.colsurfa.2009.10.029
- [66] Wang, C., Piao, C., Zhai, X., Hickman, F. N., & Li, J. (2010): *Synthesis and characterization of hydrophobic calcium carbonate particles via a dodecanoic acid inducing process*, **Powder Technology**, vol. 198(1), pp.131-135. DOI:10.1016/j.powtec.2009.10.026
- [67] Levi-Kalisman, Y., Falini, G., & Addadi, L. (2002): *Structural Differences Between Biogenic Amorphous Calcium Carbonate Phases Using X-ray Absorption Spectroscopy*, **Advanced Functional Materials**, vol. 12(1), pp.8-15. DOI:10.1002/1616-3028(2002101)12:13.0.CO;2-C
- [68] Zhao, L., Zhang, Y., Miao, Y., & Nie, L. (2016): *Controlled synthesis, characterization and application of hydrophobic calcium carbonate nanoparticles in PVC*, **Powder Technology**, vol.288, pp. 184-190. DOI:10.1016/j.powtec.2015.11.001
- [69] Levi-Kalisman, Y., Raz, S., Weiner, S., Addadi, L., & Sagi, I. (2002): *Structural Differences Between Biogenic Amorphous Calcium Carbonate Phases Using X-ray Absorption Spectroscopy*, **Advanced Functional Materials**, vol. 12(1), pp.43. DOI:10.1002/1616-3028(2002101)12:13.0.co;2-c
- [70] Patil, C. B., Kapadi, U. R., Hundiwale, D. G., & Mahulikar, P. P. (2009): *Preparation and characterization of poly (vinyl chloride) calcium carbonate nanocomposites via melt intercalation*. **Journal of Materials Science**, vol. 44(12), pp. 3118-3124. DOI:10.1007/s10853-009-3414-y
- [71] Cai, H., Yang, K., & Yi, W. (2017): *Effects of calcium carbonate on preparation and mechanical properties of wood/plastic composite*, **International Journal of Agricultural and Biological Engineering**, vol. 10(1), pp.184-190. DOI:10.3965/j.ijabe.20171001.2707
- [72] Hussain F, Hojjati M, Okamoto M, Gorga RE (2006): *Review article: Polymer-matrix Nanocomposites, Processing, Manufacturing, and Application: An Overview*, **Journal of Composite Materials**, vol. 40(17), pp.1511-1575. DOI:10.1177/0021998306067321
- [73] Chen, C., Teng, C., Su, S., Wu, W., & Yang, C. (2005): *Effects of microscale calcium carbonate and nanoscale calcium carbonate on the fusion, thermal, and mechanical characterizations of rigid poly (vinyl chloride)/calcium carbonate composites*, **Journal of Polymer Science Part B: Polymer Physics**, vol. 44(2), pp.451-460. DOI:10.1002/polb.20721
- [74] Sun, S., Li, C., Zhang, L., Du, H., & Burnell-Gray, J. (2006): *Interfacial structures and mechanical properties of PVC composites reinforced by CaCO₃ with different particle sizes and surface treatments*, **Polymer International**, vol. 55(2), pp.158-164. DOI:10.1002/pi.1932
- [75] Menard, K. P., & Menard, N. (2017). *Dynamic Mechanical Analysis*. **Encyclopedia of Analytical Chemistry**, pp. 1-25. DOI: 10.1002/9780470027318.a2007.pub3
- [76] Shimpi, N., Verma, J., & Mishra, S. (2009): *Dispersion of Nano CaCO₃ on PVC and its Influence on Mechanical and Thermal Properties*, **Journal of Composite Materials**, vol. 44(2), pp. 211-219. DOI:10.1177/0021998309344637.

- [77] May, M. A., & Saad, S. A. (2018): *Study the Effect of Temperature on Structural, Mechanical and Thermal Properties of PVC/CaCO₃ Composite*, **Iraqi Journal of Science**, vol. 59 (1A), pp.37-58. DOI:10.24996/ijs.2018.59.1a.7
- [78] Liu, C., Wu, C., & Lin, L. (2016): *A Study on the Interfacial Adhesion Strength of Different Types of Calcium Carbonate Filled Poly (vinyl chloride) Composites*, **Proceedings of the 2016 7th International Conference on Mechatronics, Control and Materials (ICMCM 2016)**. DOI:10.2991/icmcm-16.2016.61
- [79] N. Chen, C. Wan, Y. Zhang, Y. Zhang (2004): *Effect of nano-CaCO₃ on mechanical properties of PVC and PVC/Blendex blend*, **Polymer Testing**, vol. 23 (2), pp. 169-174. DOI: 10.1016/S0142-9418(03)00076-X
- [80] Kumar, P. N., Nagappan, V., & Karthikeyan, C. (2019): *Effects of Fly Ash, Calcium Carbonate Fillers on Mechanical, Moisture Absorption Properties in Poly Vinyl Chloride Resin*, **Materials Today: Proceedings**, vol. 16, pp. 1219-1225. DOI: 10.1016/j.matpr.2019.05.217
- [81] Hai, L., Lijie, D., Haiyan, X., Lipeng, W., Zhukai, L., & Chuanxi, X. (2013): *Ultraviolet light aging properties of PVC/CaCO₃ composites*, **Applied Polymer**, vol. 127(4), pp. 2749-2756. DOI: ttps://doi.org/10.1002/app.37595.
- [82] C. Pugazhendhi Sugumaran, (2015): *Experimental study on dielectric and mechanical properties of PVC cable insulation with SiO₂/CaCO₃ nanofillers*. **IEEE Electrical Insulation Magazine**, vol.31 pp.503-506. DOI:10.1109/CEIDP.2015.7352072.
- [83] Abd, A. (2014): *Studying the mechanical and electrical properties of epoxy with PVC and calcium carbonate filler*, **International Journal of Engineering & Technology**, vol. 3(4), p.545. DOI:10.14419/ijet.v3i4.3425.
- [84] Fernando, N., & Thomas, N. (2012): *Investigation of precipitated calcium carbonate as a processing aid and impact modifier in poly (vinyl chloride)*, **Polymer Engineering & Science**, vol. 52(11), pp. 2369-2374. DOI:10.1002/pen.23191.
- [85] Jazi, S. H., Bagheri, R., & Esfahany, M. N. (2015): *The effect of surface modification of (micro/nano)-calcium carbonate particles at various ratios on mechanical properties of poly (vinyl chloride) composites*, **Journal of Thermoplastic Composite Materials**, vol. 28(4), pp. 479-495. DOI:10.1177/0892705713486128.
- [86] Yang, B., Bai, Y., & Cao, Y. (2010): *Effects of inorganic nano-particles on plasticizers migration of flexible PVC*, **Journal of Applied Polymer Science**, vol. 115(4), pp. 2178-2182. DOI:10.1002/app.31310.
- [87] Liu, P., Zhao, M., & Guo, J. (2006): *Thermal Stabilities of poly (vinyl chloride)/calcium carbonate (PVC/CaCO₃) Composites*, **Journal of Macromolecular Science, Part B**, vol. 45(6), pp. 1135-1140. DOI:10.1080/00222340600962650.
- [88] Ji, Y., Xu, G., Yang, Z., Luo, H., & Tan, H. (2016): *Study on viscosity and aging process of CaCO₃ filled poly (vinyl chloride) plastisols*, **Journal of Vinyl and Additive Technology**, vol. 24, pp.53-61. DOI:10.1002/vnl.21584
- [89] Zhang, H., Chen, J.F., Zhou, H.K. (2002): *Preparation of nano-sized precipitated calcium carbonate for PVC plastisol rheology modification*, **Journal of Materials Science Letters**, vol. 21, pp. 1305-1306. DOI:10.1023/A:1016579724682
- [90] Chetanachan, W., Sutthitavil, W., & Chomcheuy, W. (2011): *Effect of nano calcium carbonate on the impact strength and accelerated weatherability of rigid poly (vinyl chloride)/acrylic impact modifier*, **Journal of Vinyl and Additive Technology**, vol. 17(2), pp. 92-97. DOI:10.1002/vnl.20266
- [91] Bystedt, J., Erlandsson, A., Enequist, B., & Josefsson, C. (1987): *The effect of small amounts of calcium carbonate on mechanical properties of rigid PVC*, **Journal of Vinyl and Additive Technology**, vol. 9(3), pp.136-139. DOI:10.1002/vnl.730090311
- [92] Kemal, I., Whittle, A., Burford, R., Vodenitcharova, T., & Hoffman, M. (2009). *Toughening of unmodified polyvinylchloride through the addition of nanoparticulate calcium carbonate*. **Journal of Polymer**, vol. 50 (16), pp.4066-4079. DOI:10.1016/j.polymer.2009.06.028
- [93] Altarazi, S. A., & Allaf, R. M. (2016): *Designing and analyzing a mixture experiment to optimize the mixing proportions of polyvinyl chloride composites*, **Journal of Applied Statistics**, vol. 44(8), pp.1441-1465. DOI:10.1080/02664763.2016.1214243
- [94] Bonadies, I., Avella, M., Avolio, R., Carfagna, C., Errico, M. E., & Gentile, G. (2011): *Poly (vinyl chloride)/CaCO₃ nanocomposites: Influence of surface treatments on the properties*. **Journal of Applied Polymer Science**, vol. 122(6), pp.3590-3598. DOI:10.1002/app.34770
- [95] Chen, N., Wan, C., Zhang, Y., Zhang, Y., & Zhang, C. (2004): *Fracture behavior of PVC/Blendex/nano-CaCO₃ composites*. **Journal of Applied Polymer Science**, vol. 95(4), pp. 953-961. DOI:10.1002/app.20786
- [96] Zhang O, Zhang C, Wu L, Sun W, Hu L (2012): *Study on Morphology and Mechanical Properties of PVC with Ultrafine CaCO₃ Surface-modified by Acrylate Macromolecular Modifiers*, **Polymers and Polymer Composites**, vol. 20(1-2), pp.191-196. DOI:10.1177/0967391112020001-236
- [97] M. Ö. Öteyaka, H. C. Öteyaka; (2019): *Chemical and mechanical properties analysis of extruded polyvinyl chlorolide (PVC)/sepiolite composite*, **Sakarya University Journal of Science**, vol. 23(4), pp.633-640. DOI:10.16984/saufenbilder.464841
- [98] T. Y. Chen, W. F. Li, J. D. Jun, J. H. Pen & J. X. Chao (2010): *Modification of nanometre calcium carbonate and its application on PVC composites in situ suspension polymerisation*, **Materials Science and Technology**, vol. 26 (7), pp.871-874.
- [99] DOI: 10.1179/174328409X463289
- [100] Xiong, Y., Chen, G., & Guo, S. (2006): *The preparation of core-shell CaCO₃ particles and its effect on mechanical property of PVC composites*, **Journal of Applied Polymer Science**, vol. 102(2), pp.1084-1091. DOI:10.1002/app.24262
- [101] YOSHINOBU, N., YUKI, F., & TAKEO, I. (1998): *Tensile Test of Poly (vinyl chloride) Filled with Ground Calcium Carbonate Particles*, **Journal of Applied Polymer Science**, vol. 70(2), pp.314. DOI:10.1002/(SICI)1097-4628(19981010)70:2<311::AID-APP11>3.0.CO;2-6
- [102] B. A. Basilia, M. E.G. Panganiban, A. A.V. C. Collado, M. O.D. Pesigan, & P. A. De yro (2007): *Study on the Functionality of Nano-Precipitated Calcium Carbonate as Filler in Thermoplastics*, **Journal of Solid Mechanics and Materials Engineering**, vol. 1(4), pp.567. DOI:10.1299/jmmp.1.564

- [103] Park, E. J., Park, B. C., Kim, Y. J., Canlier, A., & Hwang, T. S. (2018). *Elimination and Substitution Compete During Amination of Poly(vinyl chloride) with Ethylenediamine: XPS Analysis and Approach of Active Site Index*. **Macromolecular Research**, 26(10), pp.913-923. DOI:10.1007/s13233-018-6123-z
- [104] Coltro, L., Pitta, J. B., & Madaleno, E. (2013). *Performance evaluation of new plasticizers for stretch PVC films*. **Polymer Testing**, 32(2), pp.272-278. DOI:10.1016/j.polymertesting.2012.11.009
- [105] Ramesh, S., Leen, K. H., Kumutha, K., & Arof, A. (2007). *FTIR studies of PVC/PMMA blend based polymer electrolytes*. **Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy**, 66(4-5), pp.1237-1242. DOI:10.1016/j.saa.2006.06.012
- [106] Stromberg, R., Straus, S., & Achhammer, B. (1958). *Infrared spectra of thermally degraded poly(vinyl-chloride)*. **Journal of Research of the National Bureau of Standards**, 60(2), pp.147-152. DOI:10.6028/jres.060.018
- [107] Abdel-Gawad, N. M., Dein, A. Z., Mansour, D. A., Ahmed, H. M., Darwish, M. M., & Lehtonen, M. (2019). *Development of industrial scale PVC nanocomposites with comprehensive enhancement in dielectric properties*. **IET Science, Measurement & Technology**, 13(1), pp.90-96. DOI:10.1049/iet-smt.2018.527
- [108] Al-Mosawi, A. I., & Marossy, K. (2019). *Effect of extrusion speed and ATO content on rheological properties of plasticised PVC*. **IOP Conference Series: Materials Science and Engineering**, Volume 117, pp. 50509-50511. DOI:10.1088/1757-899x/613/1/012029
- [109] Park, J., Kim, M., Yoon, J., Kobayashi, F., Iwasaka, Y., Hong, C., . . . Kim, Y. (2009). *Biodegradation of diisodecyl phthalate (DIDP) by Bacillus sp. SB-007*. **Journal of Basic Microbiology**, Volume 49, pp. S31-S35. DOI:10.1002/jobm.200800297
- [110] Kusmanto, F., Billham, M., & Hornsby, P. (2008). *Polymer plasticization using supercritical carbon dioxide*. **Journal of Vinyl and Additive Technology**, 14(4), pp.163-166. DOI:10.1002/vnl.20161
- [111] Horn, D., Kranz, Z., & Lamberton, J. (1964). *The composition of Eucalyptus and some other leaf waxes*. **Australian Journal of Chemistry**, 17(4), pp. 464-474. DOI:10.1071/ch9640464
- [112] Öksüz, M., & Yıldırım, H. (2005). *Effect of calcium carbonate on the mechanical and thermal properties of isotactic polypropylene/ethylene vinyl acetate blends*. **Journal of Applied Polymer Science**, 96(4), pp. 1126-1137. DOI:10.1002/app.21555
- [113] Titone, V., Mantia, F. P., & Mistretta, M. C. (2020). *The Effect of Calcium Carbonate on the Photo-Oxidative Behavior of Poly(butylene adipate-co-terephthalate)*. **Macromolecular Materials and Engineering**, 305(10), pp. 2000358(1-7). DOI:10.1002/mame.202000358
- [114] Shan, Z., Chen, Q., Fu, Q., Feng, C., Huang, C., & Zheng, W. (2019). *TSDC measurements to analyze the electrical ageing state of large generator stator bar insulation*. **Polymer Testing**, 77, pp.105901. DOI:10.1016/j.polymertesting.2019.105901.