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Preface

We would like to present, with great pleasure, the inaugural volume-7, Issue-9, September 2021, of a scholarly journal, *International Journal of Engineering Research & Science*. This journal is part of the AD Publications series *in the field of Engineering, Mathematics, Physics, Chemistry and science Research Development*, and is devoted to the gamut of Engineering and Science issues, from theoretical aspects to application-dependent studies and the validation of emerging technologies.

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Each article in this issue provides an example of a concrete industrial application or a case study of the presented methodology to amplify the impact of the contribution. We are very thankful to everybody within that community who supported the idea of creating a new Research with IJOER. We are certain that this issue will be followed by many others, reporting new developments in the Engineering and Science field. This issue would not have been possible without the great support of the Reviewer, Editorial Board members and also with our Advisory Board Members, and we would like to express our sincere thanks to all of them. We would also like to express our gratitude to the editorial staff of AD Publications, who supported us at every stage of the project. It is our hope that this fine collection of articles will be a valuable resource for *IJOER* readers and will stimulate further research into the vibrant area of Engineering and Science Research.



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Effect of Processing Method of Kidney Beans (*Phaseolus Vulgaris*) on Carcass Quality, Organ Weight and Organoleptic Properties of Broiler

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Abstract— Effect of processing method of kidney beans (*phaseolus vulgaris*) on carcass quality, organ weight and organoleptic properties of broiler was studied using one hundred and twenty day-old chicks (Arbo acre strain). Raw, dehulled and boiled KB were used for treatment 2, 3 and 4, respectively. Prior to grinding of KB, 50kg was boiled at 100 °C for 1 hour, 10kg was dehulled by immersing in cool water for 20 hours, and seed coat removed. The work was carried out using completely randomized design, with four treatments and three replicates of 10 birds per replicate. Data collected were analyzed using SPSS version 22. The relative weight of liver, spleen and heart had no significant effect ($p>0.05$) within the dietary treatment. The weight of the liver was smaller in birds fed raw kidney beans meal and the liver was characterized by marked coagulative necrosis. The weight of the gizzard was significantly ($p<0.05$) higher in birds fed control diet. Furthermore there were no significant differences ($p>0.05$) in birds fed boiled, dehulled and control diets on the breast weight but significantly smaller in those fed raw diet meal. There were also no significant differences on the drumstick, thigh, neck and head size. Tenderness was not significantly affected by the dietary treatments. Juiciness, taste, and flavour intensity showed significant differences within the dietary treatment while overall acceptability showed a significant increase with the boiled kidney bean meal. Consequently, birds fed with BKD performed better in the organoleptic properties, while those on dehulled kidney beans meal gave better result for organ weight and carcass yield. In conclusion, sensory characteristics, organ weight, and carcass yield is an indication that kidney beans especially the boiled and dehulled one can serve as a feed ingredient in broiler ration would have no adverse effect.

Keywords— dehulled kidney beans, organoleptic properties, sensory characteristics.

I. INTRODUCTION

According to Dipeolu *et al.* (2004) poultry industry has been described as the fastest means of ameliorating the animal protein deficiency in third world countries particularly in Nigeria, due to the high turnover rate associated with poultry production and consequent economic efficiency. Feed, which accounts for 60-80% of the total cost of production of most livestock species, is by far the major factor limiting the growth and expansion of the livestock industry (Ogundipe, 1992; Ikani *et al.*, 2001). Currently, the convectional protein ingredient for monogastric animal feed production such as soyabean and groundnut cakes are often scarce and expensive due to the high demand for them for human consumption. Consequently, animal nutritionists in developing countries such as Nigeria have resorted to exploring other potential and hitherto neglected feed resources in order for the monogastric animal feed industry to have a wider range of alternatives to choose from. The availability of alternative sources of nutrient will encourage a shift to the sources for which there is less competition. Efforts have been made to use the vegetable protein sources such as pigeon pea (Amaefule and Obioha, 2001; Lorgyer *et al.*, 2009; Lorgyer, 2010), *Mucuna pruriens* seeds (Emenalom and Udedibie, 1998; Tuleun *et al.*, 2011) and Jack beans (Esonu *et al.*, 1998) in monogastric diets with encouraging results. There are however, many other legumes whose seeds can be explored

for their nutritional value for monogastric animals (Bawa, 2003); one of such legumes is the kidney beans (*Phaseolus vulgaris*).

Kidney beans (*Phaseolus vulgaris*) also referred to as common bean belong to the genus phaseolus in the legume family Fabaceae and is botanically classified as dicotyledons (Uebersax, 2006). Kidney bean is a potential feed source for pigs and poultry because of its high content of protein, energy. The amino acid profile is similar to that of soyabean, except for lower level of methionine (Laurena *et al.*, 1991).

However, like other grain legumes, the usefulness of kidney beans as a feed factor ingredient for monogastric animal may be limited due to the presence of some anti-nutritional factors which include trypsin inhibitor, hydrocyanic acid, tannin, phytic acid, oxalate and lectin (Olomu, 2011). It has been established that heat treatment and other processing methods exert beneficial effects on the nutritional quality of the seed of grain legumes by destroying the anti-nutritional factors inherent in them (Balogun *et al.*, 2001). Some of the anti-nutritional factors are, however thermostable. Thus different processing method should be applied either alone or in combination with heat treatment. This is needful because some researchers have reported that the effectiveness of heat treatment in detoxifying tannin, phytate and oxalate in kidney beans is low (Emiola *et al.*, 2007).

Thus, emphasis has been placed on the various ways of inactivating the anti-nutritional factors in the kidney bean and improvement of its nutritive value. However, little attention has been given to the evaluation of the effect of sun drying, or dehulling or aqueous heating of kidney beans (*Phaseolus vulgaris*) prior to inclusion in poultry feed. Consequently, we aimed at evaluating the effect of feeding sun dried, dehulled and aqueous heated kidney beans on the carcass quality, organ weight and organoleptic properties on broiler chicken. Specifically, the study determines:

- The weight of the heart, gizzard, liver, spleen and intestine of broiler fed raw, dehulled and aqueous heated *Phaseolus vulgaris* seed based meals.
- The carcass quality via drumstick, thigh, neck, head, breast, shank, wing of broiler fed raw, dehulled and boiled *Phaseolus vulgaris* based diet.
- The juiciness, tenderness, taste, flavour intensity and overall acceptability of broiler fed raw, dehulled and boiled *Phaseolus vulgaris* based diet.

II. MATERIALS AND METHODS

The research was undertaken at the Poultry unit of the Department of Animal Science and Technology, Nnamdi Azikiwe University, Awka, Anambra State, Nigeria. Awka is the Capital of Anambra State and it is in the tropical rainfall zone of Nigeria. The mean annual temperature, rainfall and humidity are 27.0 °C, 1828 mm and 80 %, respectively. The driest month is December with a rainfall of 7mm and the highest is September having an average of 306mm. Rainy season last for six months, occurring from April to July and September and October. It is located within the latitude of 6°12'25"N and longitude, 7°04'04"E.

The Kidney bean (*Phaseolus vulgaris*) used in the study was procured from Eke Awka Market in Awka, Anambra State. They were divided into three equal parts for the respective processing method before inclusion in the diet. For the boiled, the dry seeds were cleaned from dust and dirty materials, weighed and then poured into a cooking pot with water boiling at 100 °C and left for a period of an hour after which it was sundried for 4 days using empty rice bag before milling. For the dehulled, the dry beans were cleaned from dust and dirt materials and soaked in cool water for between 18-24 hours after which the seeds were removed using grinding machine; then the seed coats were separated from the beans. The beans were then rinsed and sundried using empty rice bag until they were sufficiently dried. The raw kidney bean together with the aqueous heated kidney beans were then grinded using a grinder.

One hundred and twenty day old broiler chicks of the breed Arbo Acre were procured from Fidan hatchery, Ibadan, Oyo State for the experiment. The birds were reared in a deep litter system, and acclimatized for one week and fed with commercial broiler starter (vital feed) bought from Eke Awka market for one week. On the 8th day, the chicks were weighed individually and distributed into the four treatment group and replicates. The treatments were: T1- Control diet; T2- Birds fed

raw kidney bean meal diet; T3 – Birds fed dehulled kidney bean meal diet; T4 – Birds fed aqueous kidney bean meal diet. So, the experimental design was a one way classification in a completely randomized design (CRD) with the following model.

$$X_{ij} = \mu + \alpha_i + \epsilon_{ij} \quad (1)$$

Where X_{ij} is the observed value of each of the response variables (carcass characteristics, organ weight and organoleptic quality characteristics) arising as a result of:

μ = the overall population mean

α_i = observed effect of the dietary treatment

ϵ_{ij} = random or residual error due to the experimentation

Brooding was done with the chicks in the four treatment group and adequate heat provided. Light was set out at night for enhanced growth. Each treatment was separated into three replica with 10 birds in each replica and the birds were fed their respective experimental diet daily. By the end of the starter phase at 4 weeks, the finisher experimental diet was given to the birds as in the starter phase. The chicks were given an anti-stress formulation in their drinking water (Milk and sugar) on the first day to relieved transportation stress. They were given Lasota vaccine on the first day of arrival. Feed and water were provided *ad libitum*. The chicks were vaccinated against Gumboro and Newcastle disease at 2 and 3 weeks of age, respectively. Prophylactic doses of coccidiostat, (Ancoban), vitamin and antibiotic were regularly provided in their drinking water at a dose of 1ml liter of water. Litters were being changed weekly.

The feeding trial was carried out in two phases; the starter and the finishes phase. 100kg starter diet were formulated for the four treatment. Diet 1 (treatment 1) with no inclusion of *Phaseolus vulgaris*, Diet 2 (treatment 2) with the inclusion of 10% raw *Phaseolus vulgaris*, Diet 3 (treatment 3) with the inclusion of 10% dehulled *Phaseolus vulgaris* and Diet 4 (treatment) with the inclusion of 10% boiled *Phaseolus vulgaris*. The finisher diet was adjusted to suit the required broiler finisher phase of lesser protein and higher carbohydrate. Thus a 500kg finisher was formulated.

Iso nitrogenous and iso calorific diets were compounded using zero percent (T1), 10 % raw (T2), dehulled (T3) and aqueous (T4) kidney bean meal.

At the end of the metabolism trial, 12 birds from each replicate of different treatments were starved over night to empty their gut contents, weighed and slaughtered. They were left to bleed completely then scalded in hot water and defeathered; their weight after defeathering and evisceration were taken. The cut up parts which include thighs, drumstick, shank, wing, neck, breast, and head were weighted and expressed as percentage of the eviscerated weight. The organ such as heart liver, spleen and gizzard were also weighed using a sensitive electronic scale.

Samples were collected from the breast of the experimental bird and cut into small size and cooked for approximately 20minutes with 5g of salt. Thereafter, they were presented in dishes under bright light to an untrained panel of 15 tasters. The panellists Include student of Unizik and staff of Ezi Awka Community Secondary School. A structural questionnaire was designed to solicit responses about juiciness, tenderness, tenderness, taste flour intensity and overall acceptability of the meat (see appendix I) using 5 point hedonic scale as described by Barylko-Pikielna (1975) with one being the least favourable of each parameter and 6 being its best condition. The scale was thoroughly explained to the panellists prior to the tasting session. Warm water was provided with which panellists rinsed their mouth between samples tasted.

Data collected were subjected to analysis of variance (ANOVA) using SPSS-22 release 7.2 statistical software. The differences between treatment means were separated using the least significant different (LSD) test.

III. RESULTS

Table 1 presents the effect of raw, dehulled and boiled kidney beans based meal on the organoleptic properties of broiler chickens.

TABLE 1
THE EFFECT OF RAW, DEHULLED AND BOILED KIDNEY BEANS BASED MEAL ON THE ORGANOLEPTIC PROPERTIES OF BROILER CHICKENS

Parameters	Treatment diets			
	T1(control)	T2(RKB)	T3(DKB)	T4(BKB)
Juiciness	3.53 ^b	3.82 ^{ab}	3.69 ^{ab}	3.95 ^a
Tenderness	3.36 ^a	3.76 ^a	3.69 ^a	3.86 ^a
Taste	3.36 ^b	3.33 ^b	3.51 ^b	4.08 ^a
Flavor intensity	3.15 ^b	3.18 ^b	3.27 ^{ab}	3.96 ^a
Acceptability	3.42 ^b	3.56 ^b	3.30 ^b	4.20 ^a

**Means bearing different superscripts along the same row were significantly different at $p < 0.05$.*

According to the Table there were no significant differences ($p > 0.05$) in the juiciness of the birds fed T2, T3 and T4. There were also no significant differences in the juiciness of birds fed T2, T3, and T1. However, there was a significant difference ($p < 0.05$) in the juiciness of birds fed T1 and T4. The result showed no significant differences ($p > 0.05$) in the tenderness and taste of the chicken fed the different treatments, except in taste of birds fed T4 and T2 ($p < 0.05$). Flavour intensity of birds fed T1, T2 and T3 were similar ($p > 0.05$), while significance difference was observed between the birds fed T3 and T4 diets. But no significant differences ($p > 0.05$) were recorded in the overall acceptability of birds, except for T4.

Table 2 presents the carcass yield of broilers fed raw, dehulled and boiled kidney bean seed meal based diet

TABLE 2
THE CARCASS YIELD OF BROILERS FED RAW, DEHULLED AND BOILED KIDNEY BEAN SEED MEAL BASED DIET

Parameters	Treatment diets			
	T1(control)	T2(RKB)	T3(DKB)	T4(BKB)
Breast	210.67 ^a	129.00 ^b	195.00 ^a	189.00 ^{ab}
Drumstick	214.00 ^a	191.00 ^b	215.00 ^a	205.67 ^a
Thigh	160.33 ^b	171.00 ^{ab}	198.33 ^a	197.33 ^a
Wings	173.33 ^{ab}	166.00 ^b	181.00 ^a	166.00 ^b
Neck	94.67 ^b	95.67 ^{ab}	100.67 ^a	103.00 ^a
Head	57.67 ^a	50.00 ^b	54.00 ^a	53.00 ^a
Shank	91.67 ^a	80.33 ^{ab}	71.33 ^b	94.67 ^a

**Means bearing different superscripts along the same row were significantly different at $p < 0.05$.*

No significant differences ($p > 0.05$) were recorded in the breast weight and drumstick of all the birds, except for bird fed RKB. Birds fed RKB were similar to all the groups ($p > 0.05$), whereas birds in control group were significantly different ($p < 0.05$) from those in DKB and BKB. Wing size was significantly higher ($p < 0.05$) in DKB than other treatment groups except for control; similarly, the size of the neck was smaller in control group than other groups except for those fed RKB. Except for birds fed RKB, the size of the head was uniform ($p > 0.05$) in all the groups. There were no significant differences ($p > 0.05$) in shank weight between birds fed control diet, BKB and RKB. There is also no significant difference in the shank weight of birds fed RKB and DKB.

Table 3 presents the organ weight of broilers fed raw, dehulled and boiled kidney bean seed meal based diet

TABLE 3
THE ORGAN WEIGHT OF BROILERS FED RAW, DEHULLED AND BOILED KIDNEY BEAN SEED MEAL BASED DIET

Parameters	Treatments			
	T1(control)	T2(RKB)	T3(DKB)	T4(BKB)
Heart	11.00±1.73 ^a	10.33±0.58 ^a	12.67±2.08 ^a	12.00±1.57 ^a
Gizzard	89.677±2.08 ^a	64.67±10.79 ^b	77.00±21.38 ^{ab}	67.00±2.65 ^{ab}
Liver	50.67±6.66 ^a	45.67±8.50 ^b	52.33±4.51 ^a	52.33±11.68 ^a
Spleen	2.00±0.00 ^a	2.00±0.00 ^a	3.00±1.00 ^a	2.33±0.57 ^a
Intestine	19.33±4.04 ^a	18.67±3.22 ^a	19.67±3.51 ^a	18.67±2.04 ^a
Live weight	2008.33±95.7 ^a	1877.33±107.83 ^a	2061.67±229.44 ^a	2000.00±52.51 ^a
Plucked weight	1887.33±163.5 ^{ab}	1678.00±23.26 ^b	1901.00±157.87 ^{ab}	1993.00±173.85 ^a

**Means bearing different superscripts along the same row were significantly different at $p < 0.05$.*

The study observed similar ($p>0.05$) heart, spleen, intestine and live weight across all the treatment groups. Smaller liver, gizzard, and plucked weight were recorded in birds fed raw kidney beans ($p<0.05$), however, the gizzard and spleen of birds fed raw kidney beans did not vary with those fed DKB.

IV. DISCUSSION

There were no significant differences on the weight of the heart, liver, spleen and intestine indicating that the three methods used in processing of KB had no deleterious effect on the internal organs of the broilers. Wafar *et al.* (2017) reported a similar result on the organs (liver and gizzard) while working with pumpkin seed based meal on the performance and carcass characteristics of broiler chickens. They maintained that the increasing level of the pumpkin seed in the diet did not affect the internal organs. Again, Soultan (2009) observed that 5% inclusion of palm kernel cake in broiler diet had no significantly effect on spleen and live weight. However, Fasuyi (2007) and Tamburawa *et al.* (2016) reported increase in weight of heart and liver while feeding some supplements.

Yakubu (2017) also gave similar result that *Jatropha curcass* seed meal increased heart weight which according to him might have been caused by increased metabolic requirement. There is also a significant difference in the weight of the gizzard across the dietary treatment. This could be as a result of extra muscular activity in breaking down ingesta which have high fiber. Richard (2012) reported a similar result and attributed a higher gizzard weight obtained to increase in frequency of contraction of this organ to reduce fiber particles. There is a significant difference between bird fed treatment diet two and other treatments. This could be as result of toxic substance. The result is in line with the report of Emiola *et al.* (2007) who equally observed that the liver was characterized by marked coagulative necrosis and degeneration of the hepatocytes of bird when fed raw and dehulled seed meal. Ortiz *et al.* (1994) also observed a degeneration of the hepatocytes in the liver which he said was due to the high tannin content of the diet.

No significant differences in the breast weight of birds fed boiled, dehulled and control diet implies that either processing method is ideal as long as breast weight is concern. Thus the treatments did not exhibit any detrimental effect on breast weight. The difference observed on the breast weight of birds fed raw kidney bean with others indicate insufficient nutrient for tissues synthesis in the group. This result correlates with that of Tuleun *et al.* (2011) who in their study reported a decreased in the breast weight of bird fed fermented *Mucuna* seed meal. He claimed that the nutrients required for tissues synthesis was not sufficient and this could be attributed to poor utilization of protein due to the presence of ant nutritional factors possibly tannin. Muhammad (2017) reported a high breast weight and better feed utilization when fed 20% pigeon pea boiled with potash diet in birds because of the improved palatability with inclusion of boiled pigeon pea seed in diet.

The drumstick, thigh, neck and head of the different dietary treatments were not significantly similar. This may be that the carcass was not influenced by the treatment. This result concurs observation of Aletor (1992) who reported from his study on the effect of different processed soybeans on the performance, organ weight, carcass yield on the economic producing broiler that the carcass were not significantly influenced by the dietary treatments. The differences observed in the shank weight and this result is in agreement with Aletor (1992).

There were no significant difference on the juiciness, tenderness and flavor intensity of the broiler fed the different dietary treatment. This may be due to the age of the bird at slaughter, adequate water in the meat and similar fat level in the meat. This result agrees with Teye *et al.* (2011). The juiciness in meat arises from the moisture released by the meat during chewing (Howard, 1976; Christens *et al.*, 2000). According to Lowrie (1976) and Ledward (2006) fat in meat improves the appearance, juiciness and other sensory qualities of meat. The taste and overall acceptability of the dietary treatment four were significantly higher than other treatment groups. Boiled kidney bean was observed to be most preferred in terms of taste, overall acceptability, flavor intensity in addition juiciness and tenderness.

REFERENCES

- [1] Aguilar, Y.M., Yero O.M, Navarro MIV, Hurtado CAB, Lopez JAC, Mejia LGB (2011). Effect of Squash Seed Meal (*Cucurbita moschata*) On Broiler Performance, Sensory Meat Quality, and Blood Lipid Profile. *Brazilian Journal of Poultry Science* .13(4):219-226.
- [2] Amao, S.R., Oyewumi, S.O., Olatunde, A.K., Oguntude, M.M. (2015). Carcass Indices and Organoleptic Properties of Organically Raised Broiler Chicken in Derived Savanna Zone of Nigeria. *Journal of Natural Science Research*. 5(20):5-10.
- [3] Arija I., Centano C, Viveros I.A, Brenes A, Marzo F, Illera J.C and Silvang G. (2006). Nutritional Evaluation of Raw and Extruded Kidney Bean (*Phaseolus vulgaris* L. Var. Pinto) in Chicken Diets. *Poultry science* 85:635-644.

- [4] Azra Yasmin, Aurangzeb, Abdulwaid Khale, Gholam Mohi-ud Din Paracha, Amal Badshah Khattak. (2008). Effect of Processing on Antinutritional Factors of Red Kidney Beans (*Phaseolus vulgaris*). *Food and Bioprocess Technology*. 1(4): PP 415-419.
- [5] Barylko-Pikielna, N. (1975). Outline of food sensory analysis. Ed. PWN, Warszawa.
- [6] Bressiani, R. and Sosa, J.L.(1990). Effects of Processing on the Nutritive Value of Jack Beans. *Plant food Human Nutrition* 40:201L-214.
- [7] Dinesh D., Jayasena, Dong UK A.H.N., Kichang Nam and Cheorun J.O. (2013). Flavour Chemistry of Chicken Meat A Review. *Asian Australian Journal of Animal Sciences* 26(5): PP 732-742.
- [8] E Ivarsson, and H. Wall (2017). Effects of Toasting, Inclusion Levels and Different Enzyme Supplementation of Faba Beans on the Growth Performance of Broiler Chickens. *The Journal of Applied Poultry Research* 26 (4): PP 467-475.
- [9] Emenalom O.O. and Udedibie A.B.I (1998). Effect of Dietary Raw, Cooked, and Toasted *Mucuna pruriens* Seeds (Velvet beans). On the Performance of Finisher Broilers. *Nigerian Journal of Animal Production*. 25:115-118.
- [10] Emiola I.A, Ologhobo A.D, Alcinlade J.A, Adedeji and Bamigbade O.M (2003). Effects of Inclusion of Differently Processed *Mucuna* Seed Meal on Performance Characteristics of Broiler. *Tropical Production Investment* 6:13-21.
- [11] Emiola, I.A., Ologhobo A.D. and Gous R.M. (2007). Performance and Histological Responses of Internal Organs of Broiler Chickens Fed Raw, Dehulled and Aqueous and Dry- Heated Kidney beans Meal. *Poultry Science*. 86:1234-1240.
- [12] Hosseini-Vashan, S.J, Golian A.R.A, Motaghinia G.H, Narnvari and Hamedi. M (2010). Comparison of Growth Performance and Carcass Characteristics of Broiler Chickens Fed Diets with Various Energy and Constant Energy to Protein Ration. *Medwell Journal* 9(20):2565-2570.
- [13] Inobeme, A, Nlemadim A.B., Obigwu P.A., Ikechukwu G., and Ajai.A.I. (2014). Determination of Proximate and Mineral Composition of White Cowpea Beans (*Vigna unguiculata*) Collected from Market Minna. *Internal Journal of Scientific and Engineering Research*. 5(8).
- [14] Leesson, S. (2008). Prediction of Commercial Poultry Nutrition. *Journal of Applied Poultry Research*. 17(10):315-409.
- [15] Muamer, M. Musa, Khalid M. Elamin, Khadiga A., Abdel Ati, Hind A.A., Elagib and Ahmed M. Musa (2012). Effects of Dietary Levels of Decorticated Cowpea (*Vigna unguiculata*) Supplemented with Molasses on Broiler Chicks Performance and Carcass Traits. *Pakistan Journal of Biological Science* 15(21):1010-1018.
- [16] Nsikan .E., Effiong E and Lydia Thompson (2016). Carcass characteristics and Internal Organs of Broiler Fed Varying Levels of Full Fat Palm Kernel Meal. *Journal of Biology, Agriculture and Healthcare*. 6(18).
- [17] Obun C. O (2008). Performance, digestibility, carcass and organ weight of finisher broiler chick fed graded levels of fermented locust beans (*Parkia biglobosa*) seed meal. *Asian journal of poultry science*. 2(1): 17-23.
- [18] Obun, C.O., Olafadehan, O. A., Ayanwale, B.A., and Inuwa, M. (2008). Growth, Carcass and Organ Weight of Broiler Fed Differently Processed *Detarium microcarpum* (Guill and Sperr). Seed meal. *Livestock Research for Rural Development*. 20(126): 8.
- [19] Onyimonyi, A.E., Adeyemi Olabode and Okeke, G. C. (2009). Performance and economic Characteristics of Broiler Fed Varying Dietary Levels of Neem Leaf Meal (*Azadirachta indica*). *International Journal of Poultry Science*. 8(3):256-259.
- [20] Pippen, E.L., Mecchi, E.P., and Nonalca, M. (1969). Origin and Nature of Aroma in Fat of Cooked Poultry. *Journal of Food Sciences*. 34(5):436-442.
- [21] Prince Chisoro, Tinashe Kaguru, Never Assan (2017). Baobab Seed as an Alternative Protein Source in poultry. *Scientific Journal of Review* 6(1): PP 509-518.
- [22] Robert Begbie, Alexander W Ross. (1993). Resistance of the kidney beans reserve protein, phaseolin to proteolysis porcine digestive tract. *Journal of the science of food and agriculture* 61(3).
- [23] Shang, Rui. Chen, Chanyou (2016). The Diversity of Four Anti-nutritional Factors in Common Beans. *Horticultural Plant Journal* (2): PP 97-104.
- [24] Soltan M.A. (2009). Growth performance, Immune Response and Carcass Traits of Broiler Chicken Graded Levels of Palm Kernel Cake without or with Enzyme Supplementation. *Livestock Research Development* 21(3).
- [25] Taju Hussein, Mengisturge, Getachow Animut (2015). Effect of Replacing Soy bean meal (*Glycine max*) with Kidney beans (*Phaseolus vulgaris*) on Egg Quality Parameter of white leghorn layer. *International journal of livestock production* 7(6): PP 33- 40.
- [26] Tamburawa, M.S., Ogundipe, S. O., Tegbe, T.S.B., And Olugbemi, T. S. (2016). Effect of Dietary Utilization of Seed Meal of Cooked African locust bean (*Parkia biglobosa*) in Broiler Chickens. *Journal of Animal Science and Veterinary Medicine* 1. pp 100-107.
- [27] Teye M, Apori S.O., and Ayeida A.A.A. (2015). Carcass Parameters and Sensory Characteristics of Broiler Chicken Fed Diets Containing Palm (*Elaeis guineensis*) Kernel oil residue. *International Journal of Current Microbiology and Applied sciences* .4(6): 1030-1038.
- [28] Tsado, D.N and Akinwolere (2015). Performance and Organoleptic properties of broiler fed Replacement Levels of Rumen Filtrate Fermented Shea nut (*Vitellaria paradoxa*) Meal for Groundnut meal. *Net Journals*.
- [29] Uchegbu M.C, Okoli I.C, Anyanwu C.E, Beetuk E, Esonu B.O and Udedibe A.B.I (2004). Performance, carcass and organ characteristics of finisher broiler fed graded levels of raw *Napolea imperialis* seed meal. *Livestock Research for Panel Development* 16(6) .
- [30] Wafar, R.J., Hannison M.I., Abdullahi. U and Makinta. A (2017). Effect of pumpkin (*Cucurbita pepo* L.) seed meal on the performance and carcass characteristics of broiler chickens. *Asian Journal of Advances in Agricultural Research*. 2(3): 1-7.
- [31] Yakubu, B, Mbahi T.F and Adama I.S. (2017). Effects of feeding raw and differently processed pigeon pea (*Cajanus cajan* (1) Millsp) seed meal on growth performance of wearier pigs. *International Journal of Agriculture and Environmental Research* 3(2).

UHPLC-DAD-ESI-MS/MS of Ethanolic Extract of the Flowers from *Spathodea Campanulata* Beauv. in Cuba

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Abstract— A sample of ethanolic extract of the flowers from *Spathodea campanulata* Beauv. was scrutinized using a UHPLC-DAD-MS/MS Thermo scientific Dionex Ultimate 3000 RS coupled to a Thermo scientific LTQ XL mass spectrometer. In addition, about nine different chemical compounds were tentatively identified in this species based on chromatography retention time (Rt), UV and MS/MS spectra and compared with those of isolated authentic compound and literature data in our country for the first time. Information obtained from these studies can be used as biomarkers in the identification and standardization of this flower as an herbal remedy and also towards monograph development on the plant.

Keywords— UHPLC, Flowers, Chemical compounds, Ethanolic extract, *Spathodea campanulata*.

I. INTRODUCTION

Spathodea campanulata P. Beauvais tree is native to Africa. In tropical Africa it is planted as an ornamental plant, e. g. in Cape Verde, Zimbabwe and Madagascar. It is widely grown in tropical and subtropical regions outside Africa [1]. This plant is also commonly found in India as an ornamental plant. This plant is also called as African tulip tree or Flame tree, *Spathodea campanulata* P. Beauv is the botanical name. It is a single species of the monotypic genus *Spathodea* in the flowering plant family Bignoniaceae which is composed of around 800 species distributed in 112 genera [2].

The flowers are used as diuretic and anti-inflammatory, while the leaves are against kidney diseases, urethra inflammations and as an antidote against animal poisons [3]. Several phytochemical studies were performed with different parts of *Spathodea campanulata* including stem barks, leaves, flowers and fruits. The leaves contain spathodol, caffeic acid, other phenolic acids and flavonoids, while fruits contain polyphenols, tannins, saponins and glycosides [4]. The plant leaves reported to have antiplasmodial, analgesic, anti-inflammatory and anti-larvicidal activity. The stem bark decoction of *Spathodea campanulata* have been displayed hypoglycemic, anti-complementary, antimalarial and anti-HIV activity [5].

The large, orange-red flowers are arranged in dense clusters (8-10 cm long) at the tips of the branches. The reddish-orange colored petals are also fused together and are shaped somewhat like a tulip flower i.e. tubular (Figure 1). Flowering occurs throughout the year, but usually peaks during spring [6].



FIGURE 1: Flowers of *Spathodea campanulata* Beauv

Preliminary phytochemical screening of *S. campanulata* revealed the presence of alkaloids, reducing sugars, carbohydrates, flavones, glycosides, and phenolic compounds [7]. Several compounds have been isolated from different parts of the plant. From the flowers has been isolated 1,1-diethoxy-3-methyl-butane, N-hexadecanoic acid, 1,2-benzenedicarboxylic acid diisooctyl ester, and oleic acid [8]. Phytol, α -methyl cinnamaldehyde, β -sitosterol-3-acetate, naringenin, catechin-3- O - α -rhamnopyranoside and 5, 6, 4'-trihydroxy flavonol-7- O - α -rhamnopyranoside, and anthocyanins [9].

The floral nectar contains a complex mixture of triterpenoids and steroids [Rev. Bras. Pl. Med., 2012. (10)]. The aim of this study therefore, was to isolate and characterize the active phytochemical(s) of *S. campanulata* flowers ethanolic extract.

II. MATERIAL AND METHODS

2.1 Sample Collection and Processing

The flowers were freshly collected in the morning between 9 and 10 AM during five days in April of 2018 in the gardens of the Faculty of Pharmacy and Foods at Havana University. Authors are waiting for identification at the herbarium of National Botany Garden of Havana, where the voucher specimen number will be deposited.

2.2 Extract and Samples Preparation

The flowers used were dried in an oven with controlled temperature at 40°C, during 5 days. The extracts were prepared with the ground material (100 g), using a Soxhlet apparatus and 95% ethanol (675 mL) for 20 hours, by triplicated. The ethanolic extracts were concentrated and rotoevaporated under vacuum to 200 mL at 120 rpm, 70°C, and 500 mbar. The concentrated extract was put on to the Lab table at room temperature until an abundant solid appear and it was recuperated by filtration.

2.3 UHPLC-DAD-ESI-MS/MS Procedures, Instrumentation, and Parameters

The LC system consisted of an UPLC/DAD/MS Thermo scientific Dionex Ultimate 3000 RS with quaternary pump, autosampler, DAD (diode array detector) Dionex with a UV-VIS at 200 nm (UV1), 250 nm (UV2), 280 nm (UV3), and 330 nm (UV4) coupled to a mass spectrometer Thermo scientific LTQ XL with ESI (ion trap analyzer) in negative ionization mode. Conditions of detection were optimizing with a Tune archive based on the behavior of quercetin. Temperature: 225°C, Voltage 5 KV, Capillary voltage 50 V. Column: Accucore RP-MS (100 $\times$ 2.1 mm $\times$ 2.6 μ m). Temperature: 35°C. Chromatographic system: eluent CAN (5%); HCOOH (0.1%). Isocratic 20 min. Flow: 0.4 mL/min (Table 1). Nitrogen gas flow: 34, auxiliary gas: 16, barrier gas: 3. Induced fragmentation gas: Helium. Was realized an experiment Full Scan in independent mode to identify the principal ions (TIC) and get the MS and MS². The sample was dissolved in methanol HPLC grade at 1 mg/mL filtered by a nylon filter of 0.20 μ m. Injection volume: 2 μ L. Mass scan between 200 - 700 u.m.a.

TABLE 1
CHROMATOGRAPHIC CONDITIONS USED IN THE STUDY

Time (min)	ACN	Formic acid 0,1 %
0	12	88
2	27	73
3	27	73
5	30	70
6	30	70
9	35	65
10	95	5
11	12	88
17	12	88

III. RESULTS AND DISCUSSION

3.1 MS-ESI Identification of phytochemicals in *S. campanulata* flowers from Cuba

Several phytochemical components in *S. campanulata* flowers were detected and tentatively identified using UHPLC with UV-visible data and ion trap electrospray mass spectrometry (IT-ESI-MS). Figure 3 shows the TIC chromatogram fingerprint at 250 nm. All of them were analyzed in negative ion mode between 0.17 and 11.38 min of retention time.

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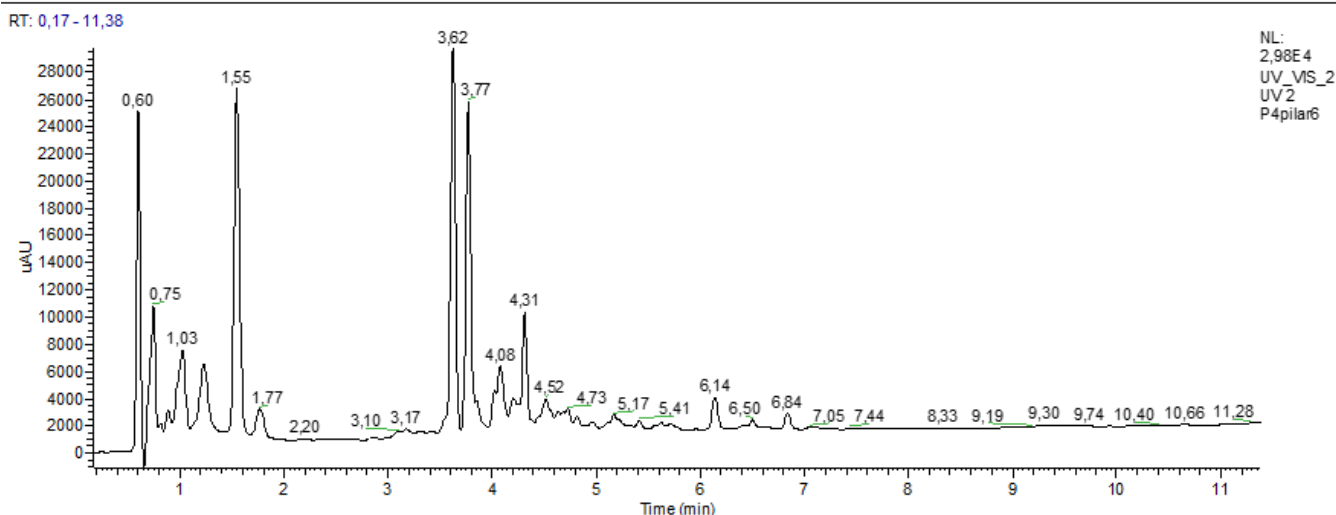


FIGURE 3: Current chromatogram at 250 nm of ethanolic extract from *S. campanulata*.

Fifteen chemical components were detected after sample running, but among them, only 9 compounds were tentatively identified, mainly glycosylated flavones, glycosylated flavonols and alkaloids (Table 2). Below is the detailed explanation and identification of the compounds using UHPLC and DAD-ESI and MSⁿ analysis.

TABLE 2
HPLC-DAD-ESI-MSⁿ DATA OF *S. CAMPANULATA* ETHANOLIC EXTRACTS

Peak No.	Retention time	UV Max	[M-H]	Other ions	Identification
1	0.61	322, 208	—	—	Unknown
2	0.74	296, 206	—	—	Unknown
3	1.02	218, 258	—	—	Unknown
4	1.22	326, 301, 247	—	—	Unknown
5	1.54	256	—	—	Unknown
6	1.77	318, 246, 222	—	—	Unknown
7	3.62	260	481	319, 205	Flavonoid glucoside
8	3.78	327, 245	523	361, 179, 163	Flavonoid glucoside
9	4.31	315, 247	523	361, 179, 163	Flavonoid glucoside
10	4.14	—	593	327, 285, 257	Flavonoid glucoside
11	4.54	—	507	345, 231, 163	Flavonoid glucoside
12	5.02	—	507	345, 231, 163	Flavonoid glucoside
13	6.65	—	582	462, 342	Alkaloid
14	6.88	—	582	462,342	Alkaloid
15	7.09	—	612	492, 466 (462), 342	Alkaloid

**According to the data reported by the technician.*

3.2 Flavonoids and Derivatives

Explanation was done taking into account the retention times, spectral data and the grouping order in which the phytochemicals were reported by the technician. The three first chemical components were registered at 3.66; 3.81 and 4.35 minutes of retention time, respectively, as is showed in Figure 4, two of them with a molecular mass of 523 Da, but with different retention times, at 3.81 and 4.35 min, respectively, indicating that both chemical components are closely related or perhaps they are isomers.

The first compound detected at 3.66 min have a molecular mass of 481 Da, having a similar molecular mass of flavonoids related with some of the four gossypetin glycosylated derivatives isolated and characterized from spices that belong to *Hibiscus* and *Talipariti* genders, but the confirmation is still pendant by NMR spectroscopy (data not shown here). We are assuming that only two aglycon moieties in nature have a MM of 318, there are gossypetin and hibiscetin. The first one is a flavonol and the second one a flavone.

As is known, four gossypetin glucosides have been isolated and characterized from different plants like *Hibiscus vitifolius* and *Hibiscus sabdariffa* L. (Gossypetin-8-O-glucoside or gossypin); from *Hibiscus sabdariffa* L., *Talipariti elatum* (Sw.) and *Talipariti tiliaceum* (Sw.) (Gossypetin-7-O-glucoside or gossypitrin or gossypetrin); from *Hibiscus sabdariffa* L. and also from *Talipariti tiliaceum* (Sw.) (Gossypetin-3-O-glucoside or gossytrin) and finally from *Abelmoschus manihot* (initially *Hibiscus manihot*) and *T. elatum* (Sw.) in Martinica and Cuba (Gossypetin-3'-O-glucoside) (11, 12, 13).

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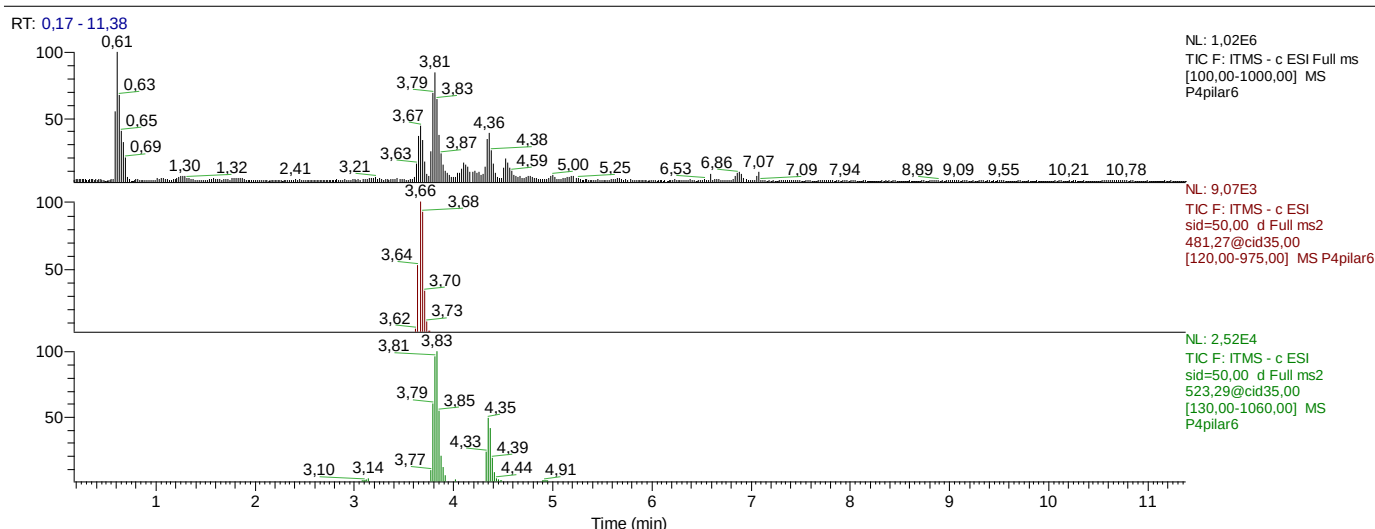


FIGURE 4: Full mass chromatogram of the three first chemical components registered.

Beside the retention time, both chemical compounds with the same molecular masses of 523 Da, showed different fragmentation pathway suggesting the presence of two isomeric forms from the same chemical component as is represented in Figure 5.

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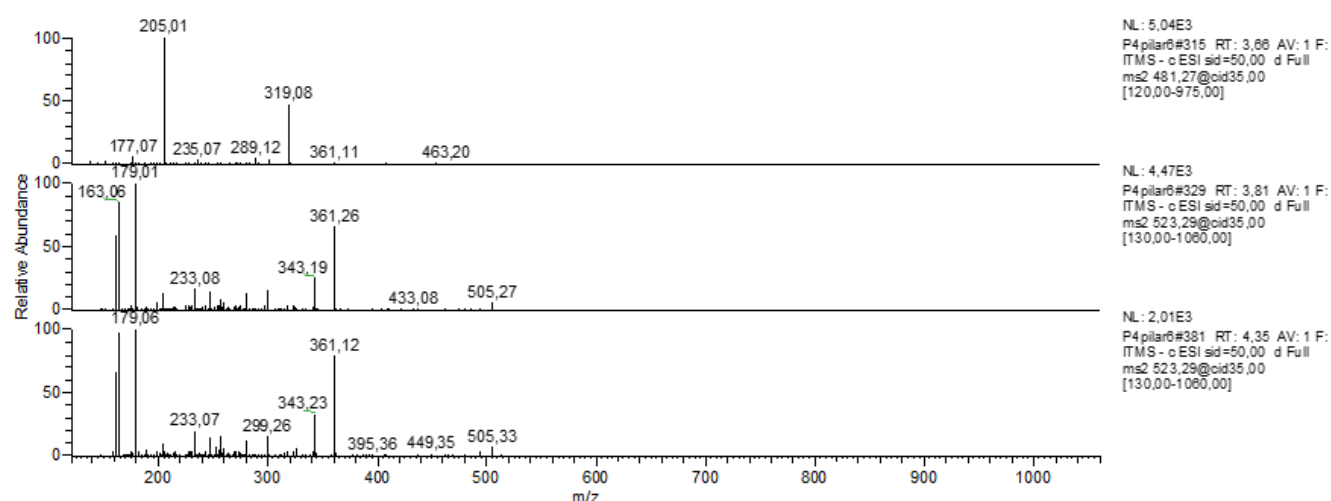


FIGURE 5: MS/MS spectrums of the three first chemical components registered.

Both compounds with MM of 507 amu have a little different behavior in their fragmentation pathway, but with the same daughter ions at m/z 345, 231 and 163. Seems to indicate that they are glucoside derivatives from 5,7-dihydroxy-3',4',5'-trimethoxyflavone with m/z 344 and $[M-H]^-$ 343, with the glucose moiety in C8 in both isomeric forms: α and β . So, we are

proposing the structure of 8- α -D-glucopiranosyl-5,7-dihydroxy-3',4',5'-trimethoxyflavone ($[M]^+$ 524; $[M-H]^-$ 523) or instead, the structure of 8- β -D-glucopiranosyl-5,7-dihydroxy-3',4',5'-trimethoxyflavone ($[M]^+$ 524; $[M-H]^-$ 523).

Three second chemical components were then registered between 4.14 and 5.02 min, keeping the same behavior like previous case, the first one of them at 4.14 min with a molecular mass of 593 Da, following by another two compounds at 4.52 and 5.02 min, respectively, but with the same molecular masses of 507 Da and different fragmentation pathway, so we can consider that both phytochemicals are isomers too (Figures 6 and 7).

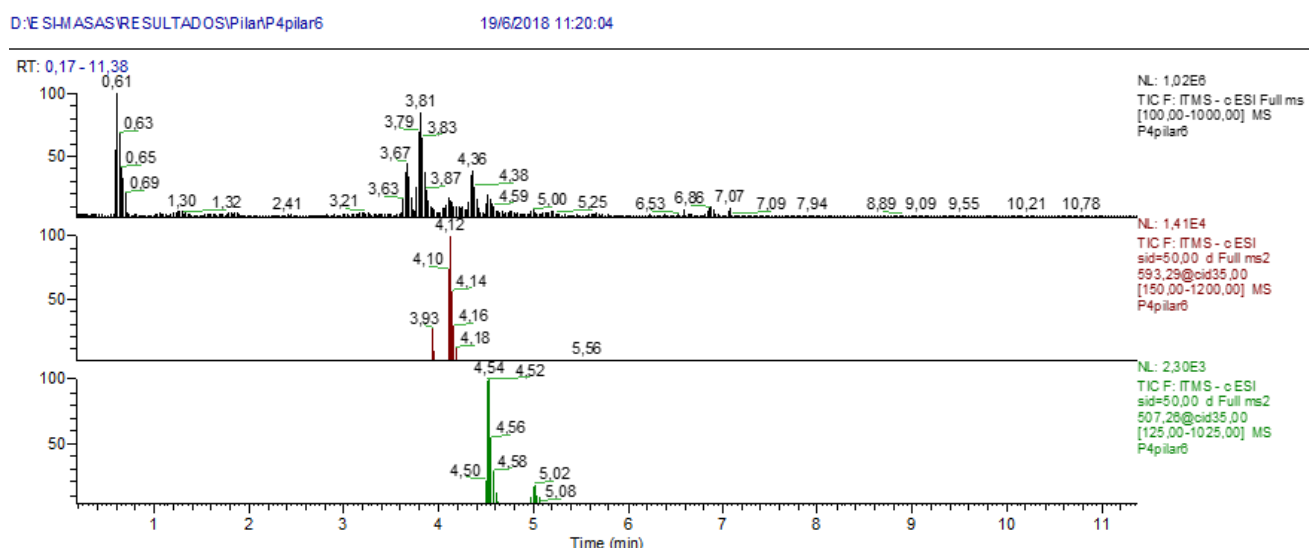


FIGURE 6: Full mass chromatogram of the three second chemical components registered.

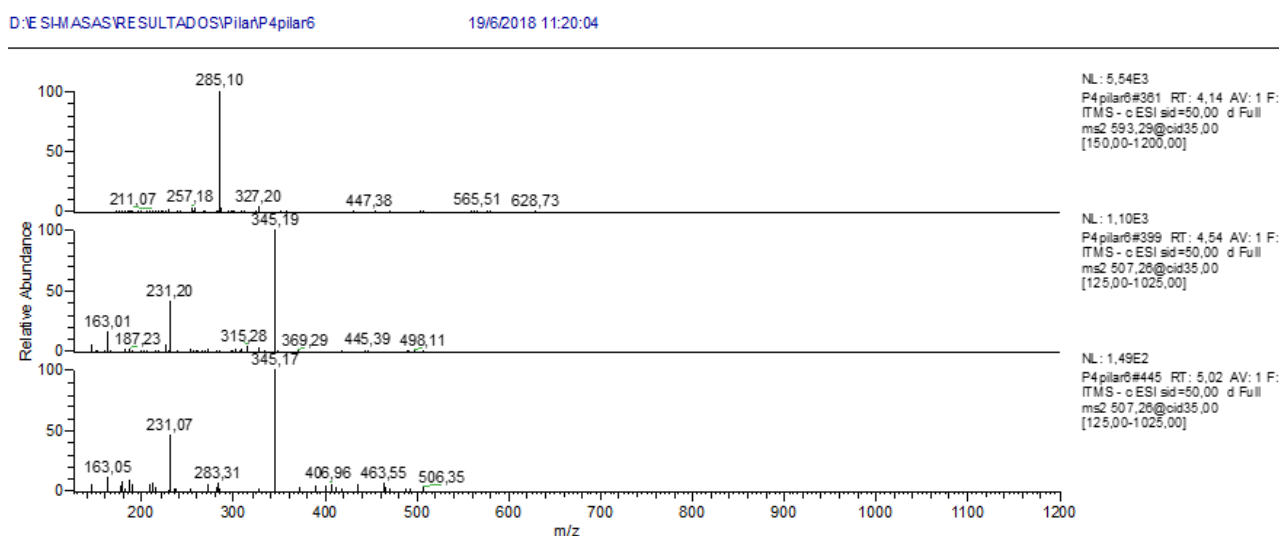


FIGURE 7: MS/MS spectra of the three second chemical components registered.

Four different secondary metabolites in nature produce a parent ion at m/z 593: luteolin-7-*O*-rutinose, kaempferol-3-*O*-rutinoside, kaempferol-3(*p*-coumarylglucoside) and glycosylated sitosterol, this one, identified in the bark and flowers extracts of *S. campanulata* by Heim et al., 2012. According to Rodriguez et al., 2009 [14], we are tentatively proposing the structure of kaempferol-3(*p*-coumarylglucoside) for the component at 4.14 min of retention time, because of its present the same fragmentation pathway of that phytocompound analyzed and daughter ions at m/z 447 and m/z 285 $[M-H-coumarylglucoside]^-$.

Both chemical components with parent ion at m/z 507 are closely related with 3-hydroxy-4',5,7- trimethoxyflavone that have a parent ion at m/z 328 to be the precursor moiety to those compounds. In such case, both structures keeping the stereochemical configuration α or β , but in this case, having one OH group attached at the flavone moiety. They presented a pseudomolecular ion at m/z 507, which experienced a hexoside loss (162 u) to produce a flavone ion at m/z 327 and, thus, was identified as 8- α -D-*O*-glucosyl-3-hydroxy-4',5,7-trimethoxyflavone (or its isomeric form β).

3.3 Alkaloids

Finally, the three third chemical components were detected at 6.65; 6.88 and 7.09 minutes, respectively. The first and second one with a molecular mass of 582 Da, probably two isomeric forms of the same phytochemical compound, and the third one with a molecular mass of 612 Da (Figures 8 and 9).

Obviously, those three phytocompounds perhaps belong to alkaloids group due to their molecular masses, according to Brindha et al., 2012, who previously reported that preliminary phytochemical screening of *Spathodea campanulata* P. Beauvais revealed the presence of alkaloids.

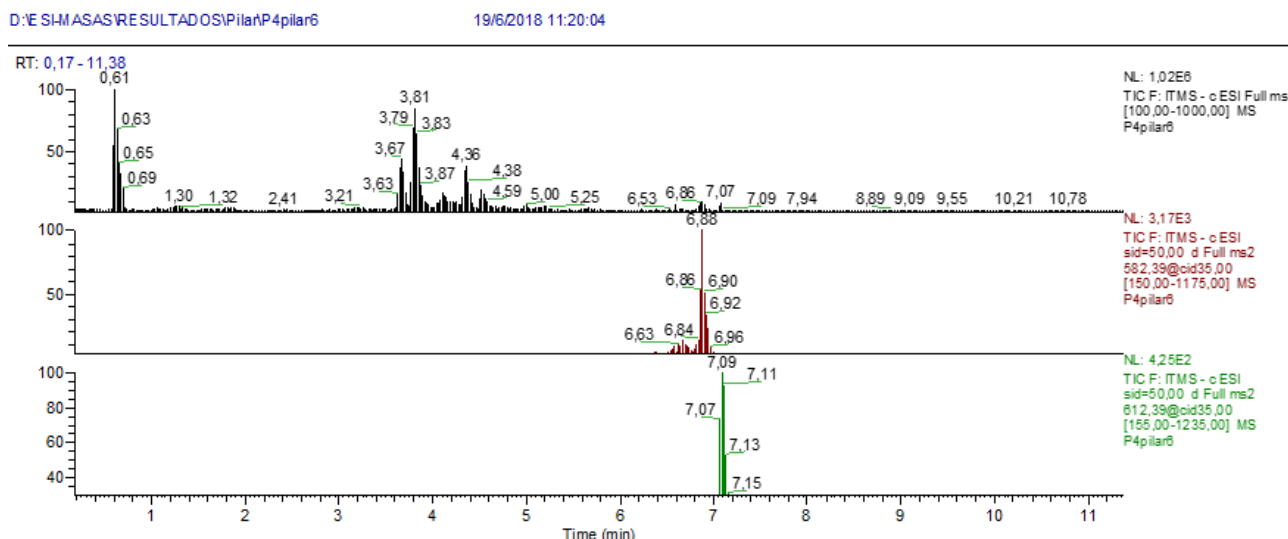


FIGURE 8: Full mass chromatogram of the three third chemical components registered.

Compounds with pseudomolecular mass at m/z 582, producing daughter ions at m/z 462 and 342, exhibit the same molecular formula $C_{35}H_{38}N_2O_6$, $[M]^+$ m/z 582. They both carry a secondary amino function. According to Fournet et al., 1987 [15], two *bis*-benzylisoquinolines were isolated and characterized from *Abuta pahni*, belonging to the genus *Abuta* (Menispermaceae, Anomospermae) spreads widely throughout tropical America. *A. pahni* is part of Amazonian curare mixtures.

The parent ion at m/z 582 are in correspondence with those both chemical components found out in this research but more experiments will be needed to confirm their presence in ethanolic extracts from the flowers of *S. campanulata* in our country.

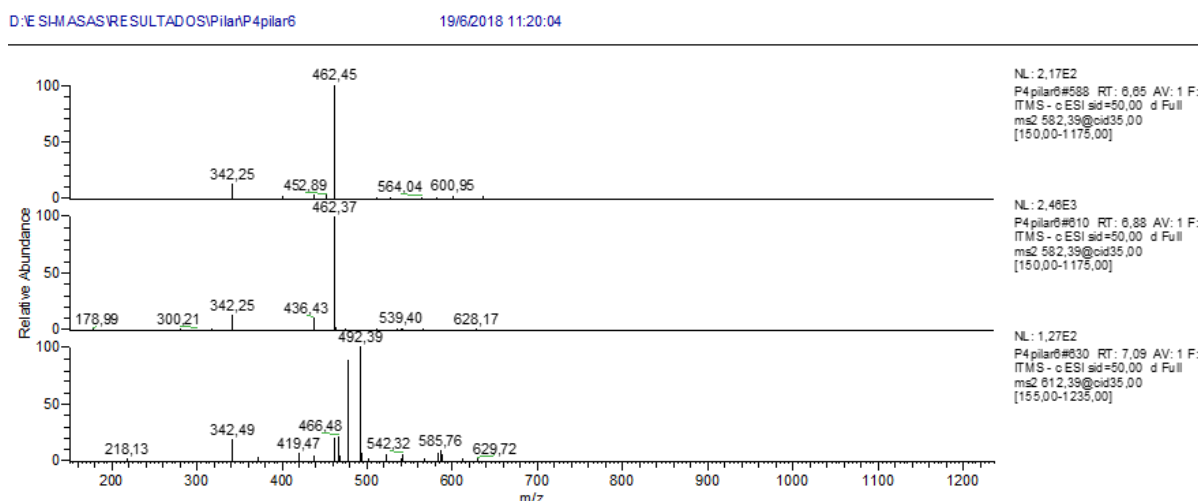
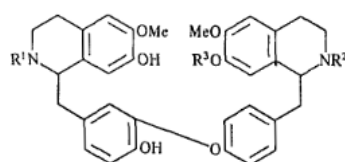


FIGURE 9: MS/MS spectrums of the three third chemical components registered.

Figure 10 shows the structures of both *bis*-benzylisoquinolines. They are in correspondence with structures 4 and 5, identified by comparison with authentic samples and the respective positions of the secondary amino and tertiary amino groups in alkaloid 4 are determined through NOE measurements (cf. values in the Experimental). It can then be given the structure 2-*N*-methyllindoldhamine. Consequently, alkaloid 5 corresponds to 2'-*N*-methyllindoldhamine.



- 1 $R^1 = \text{Me}, R^2 = \text{Me}, R^3 = \text{Me}$
 2 $R^1 = \text{Me}, R^2 = \text{H}, R^3 = \text{Me}$
 3 $R^1 = \text{H}, R^2 = \text{H}, R^3 = \text{H}$
 4 $R^1 = \text{Me}, R^2 = \text{H}, R^3 = \text{H}$
 5 $R^1 = \text{H}, R^2 = \text{Me}, R^3 = \text{H}$
 6 $R^1 = \text{Me}, R^2 = \text{Me}, R^3 = \text{H}$

FIGURE 10: Structural representation of *bis-benzylisoquinolines* from *Abuta pahni*.

Surprisingly, another two chemical components belonging to alkaloid group are in correspondence of those molecular masses: ergotamine (m/z 582) and ergocryptine (m/z 610). They were found in Ergot (*Claviceps purpurea*). Ergot alkaloids are mycotoxins generated by grass and grain pathogens such as *Claviceps*, for example. Ergot alkaloid-poisoning syndromes, such as tall fescue toxicosis from endophyte-infected tall fescue grass, are important veterinary problems for cattle, horses, sheep, pigs and chickens, with consequent impact on food, meat and dairy industries. Damage to livestock is of the order of a billion dollars a year in the United States alone (Lehner et al., 2005 [16]).

HPLC with UV and fluorescence detection are the predominant means of ergot alkaloid. Lehner et al., 2005, demonstrated the facility of using electrospray(+) mass spectrometry with multiple reaction monitoring (MRM) detection during chromatographic examination of ergot alkaloid standards of lysergic acid, lysergol, ergonovine, ergovaline, ergotamine, ergocornine, ergocryptine and ergocryptine by HPLC.

Figure 11 shows the general structure of Ergot alkaloids. The ergocryptine dehydrate was studied with settings similar to those of parental ergocryptine.

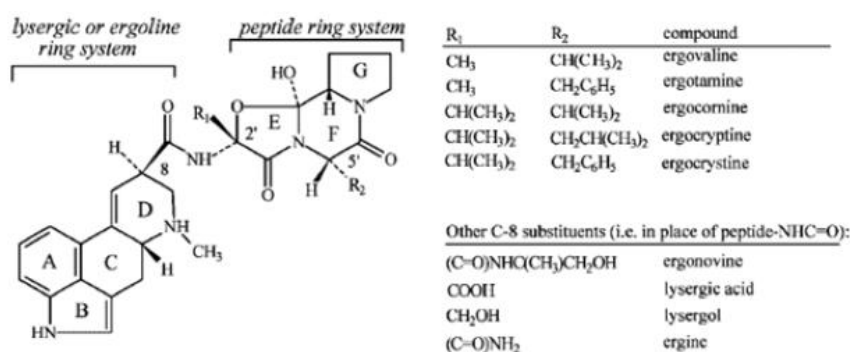
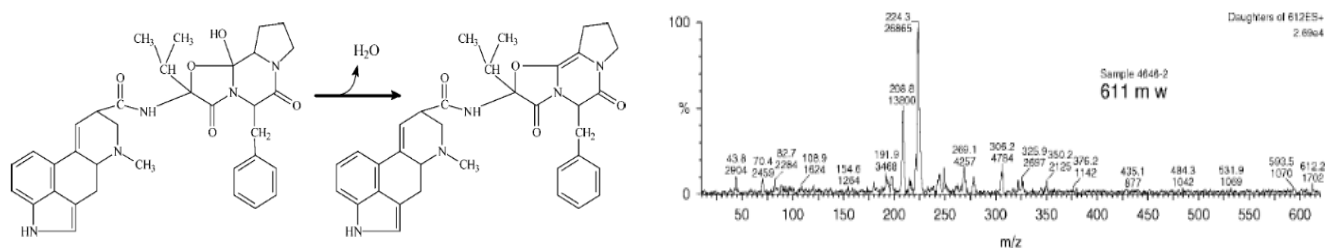


FIGURE 11: Ergot alkaloid general structure, including stereochemistry and crucial position numbering and ring labeling (rings A–D constitute lysergic or ergoline; E–G, peptide ring systems). R1 and R2 alkyl and aralkyl substituents are listed in tabular form with corresponding compound names (taken from Lehner et al., 2005).

According with that, our results are in concordance with those authors when explained the presence of ergocryptine (m/z 612) and its dehydrated form (dyhydroergocryptine, m/z 592) due to the loss of 18 u.m.a. [M-18] (Scheme 1).



SCHEME 1: Dehydration of the ergocryptine 12'-hydroxyl group in the peptide ring system (from Lehner et al., 2005)

Mass spectrum of chemical compound detected at 7.07 min shows that the phytochemical identified in *S. campanulata* in Cuba have the same fragmentation pathway of that one found in perennial ryegrass (*Lolium perenne*) and tall fescue grass (*Festuca arundinacea* Schreb) (Figure 12). For that reason, we are proposing the structure of ergocryptine that showed a $[M - H]^-$ ion at m/z 612 and MS^1 ion at m/z 592 ($[M - H - H_2O]^+$) that belongs to the dehydrated product dyhydroergocryptine.

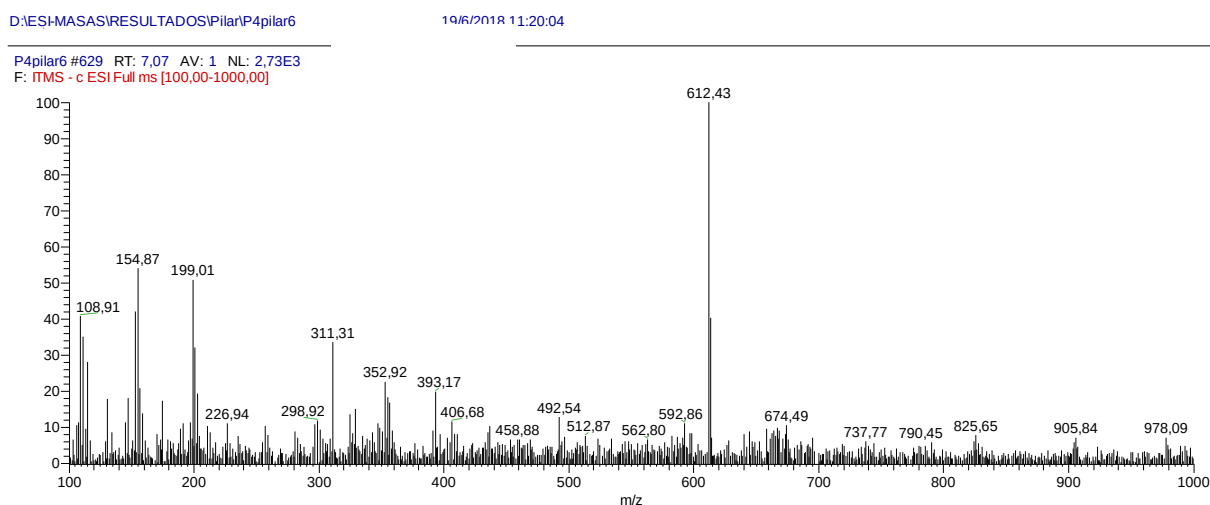


FIGURE 12: Full mass spectrum of compound with m/z 612 at 7.07 min.

Probably, degradation products in the spectrometric measurement carry out to obtain another ergot alkaloid component: ergocornine, after the loss of 30 u.m.a ($[M-30]^+$, m/z 562). Those losses are typically of alkaloids (NH_2CH , CH_2O , NO). Obviously, parent ion at m/z 582 $[M-H]^+$ could be assigned to 9,10-dyhydroergotamine or ergotamine (m/z 581), and consequently, the peak at m/z 462 correspond to ergocornine after the same loss of 30 u.m.a.

Summarizing, Table 3 relate the chemical compounds proposed to be present into ethanolic extracts of flowers from *S. campanulata* in our country. Further experiments will be done to increase the information about the existence of such phytochemicals in ethanolic extracts of the flowers of this plant, including the use of NMR spectroscopy.

TABLE 3
PROPOSAL COMPONENTS IN ETHANOLIC EXTRACTS OF THE FLOWER FROM *S. CAMPANULATA*

Compound	Ret. time	Parent ion	Daughter ions	Identification
1	3.62	481	319, 205	Gossypetin glucosides derivatives or Hibiscetin-3-O-rhamnopyranoside*
2	3.78	523	361, 179, 163	8- α -D-glucopyranosyl-5,7-dihydroxy-3',4',5'-trimethoxyflavone
3	4.31	523	361, 179, 163	8- β -D-glucopyranosyl-5,7-dihydroxy-3',4',5'-trimethoxyflavone
4	4.14	593	327, 285, 257	Kaempferol-3-(<i>p</i> -coumaroyl)glucoside)
5	4.54	507	345, 231, 163	8- α -D-glucopyranosyl-3-hydroxy-4',5,7-trimethoxyflavone
6	5.02	507	345, 231, 163	8- β -D-glucopyranosyl-3-hydroxy-4',5,7-trimethoxyflavone
7	6.65	582	462, 342	2-N-methylindolamine *9,10-dyhydroergotamine
8	6.88	582	462,342	2'-N-methylindolamine *9,10-dyhydroergotamine
9	7.09	612	592, 562, 466, 342	Ergocryptine

*more probably expected.

IV. CONCLUSIONS

The present study point outs the essentiality of collecting similar data for different plants and their flowers, as well as other parts. For the first time in our country nine different chemical compounds were tentatively identified using UHPLC-DAD-ESI-MS/MS experiments. Six of those components belong to flavonoid group (flavones and flavonol subgroup) and 3 of them to alkaloid group. All stretching related with biological activities associated to this part of the plant (antioxidant, anti-

solar, diuretic and anti-inflammatory activity). Further investigation to explore the therapeutic action of the individual phytochemicals and their mechanism of action can be encouraged.

CONFLICT OF INTEREST

Authors declare that we have not conflict of interest.

REFERENCES

- [1] Bosch CH. *Spathodea campanulata* P. Beauv. [Internet] Record from PROTA4U. Oyen, L. P. A. and Lemmens, R. H. M. J. (Editors); 2002.
- [2] Spangler RE, Olmstead RG. Phylogenetic analysis of Bignoniaceae based on the cp DNA gene sequences *rbcl* and *ndhF*. *Ann Mo Bot Gard* 1999; 86:33-46.
- [3] Mensah AY, Houghton PJ, Dickson RA, Fleischer TC, Heinrich M, Bremner P. In vitro evaluation of effects of two Ghanaian plants relevant to wound healing. *Phytother Res* 2006; 20:941-4.
- [4] Ngouela S, Tsamo E, Sondengam BL. Extractives from Bignoniaceae: constituents of the stem bark of *Spathodea campanulata*. *Planta Med* 1988; 54:476.
- [5] Niyonzima G, Laekernan G, Witvrouw M, Van Poel B, Pieters L. Hypoglycemic, anti-complement and anti-HIV activities of *Spathodea campanulata* stem bark. *Phytomedicine* 1999; 6:45-9.
- [6] Parul T, Aditi S. Natural resources from plants in the treatment of cancer: an update. *Asian J Pharm Clin Res* 2017; 10:13-22.
- [7] Brindha P, Nagarajan A, Saralla RP, R Narendran R, Sridharan K. A study on chemical and botanical standards of a traditional drug source *Spathodea campanulata* Beauv. *Int J Pharm Sci* 2012; 4:157-60.
- [8] Kumaresan M, Palanisamy PN, Kumar PE. Chemical investigation of the flower of *Spathodea campanulata* by GCMS. *J Nat Prod Plant Resour* 2011; 1:14-7.
- [9] Naglaa GS, Hanaa HE, Soheir M. Bioactivity and composition of the flowers of *Spathodea campanulata* p. Beauv. *World J Pharm Res*, 2014;3:213-30.
- [10] Heim, S.C., Guarnier, F.A., Ferreira, D.T., Braz-Filho, R., Cecchini, R., Cecchini, A.L. Antioxidant activity of *Spathodea campanulata* (Bignoniaceae) extracts. *Rev. Bras. Pl. Med.*, v.14, n.2, 2012;14:287-92.
- [11] Yaque, J.G., Cuéllar, A., Massi, L., Monan, M., Nossin, E. and François-Haugrin, F. (2016) Isolation and Characterization of Flavonols by HPLC-UV-ESI-MS/MS from *Talipariti elatum* S.w. *American Journal of Plant Sciences*, 7, 1198-1204. <http://dx.doi.org/10.4236/ajps.2016.78115>.
- [12] François-Haugrin F., Monan M., Nossin E., Smith-Ravin J. and Odile Marcelin. Antioxidant activity of an isomer of gossypitrin (gossypetin-3'-O-glucoside) isolated in the petals of *Talipariti elatum* Sw., and determination of total phenolic content of the total flower. *Journal of Pharmacognosy and Phytochemistry* 2016; 5(5): 200-208.
- [13] Yaque, J.G., Cuéllar, A., Gaysinski, M., Monan, M., Nossin, E. and François-Haugrin, F. (2016) New Reported Flavonol Characterized by NMR from the Petals of *Talipariti elatum* S. w. in Cuba. *American Journal of Plant Sciences*, 7, 1564-1569. <http://dx.doi.org/10.4236/ajps.2016.711148>.
- [14] Rodriguez I. C. et al. Medina Direct characterization of aqueous extract of *Hibiscus sabdariffa* using HPLC with diode array detection coupled to ESI and ion trap. *MS J. Sep. Sci.* 2009, 32, 3441-3448.
- [15] Pascale D., Weber J-F., Fournet A., Cavé A., and Brutenon J. Bis-benzylisoquinolines alkaloids from *Abuta pahnii*. *Phytochemistry*, Vol. 26, No. 7, pp. 2136-2137, 1987.
- [16] Lehner F. A., Craig M., Fannin N., Bush L., and Tobin T. Electrospray [+] tandem quadrupole mass spectrometry in the elucidation of ergot alkaloids chromatographed by HPLC: screening of grass or forage samples for novel toxic compounds. *J. Mass Spectrom.* 2005; 40: 1484-1502.

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

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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 $0.7 - 100 \mu\text{m}$.

2.1.6 SOCAL 312 CaCO_3

Ultrafine calcium carbonate is near to nano size. The particle size is below $0.01 \mu\text{m}$.

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.

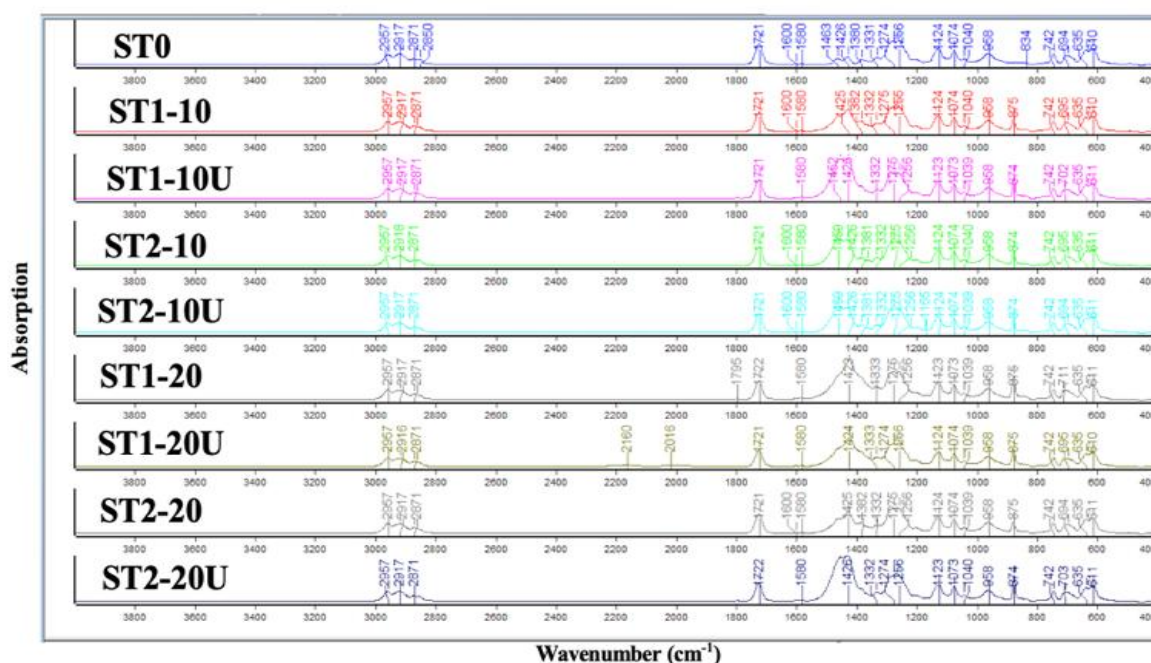


FIGURE 1: FT-IR spectras result for PVC samples.

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 CH_3 stretching and at the peaks of $2916\text{--}1918\text{ cm}^{-1}$ can be observe the CH_2 stretching which is a behavior of PVC polymer [80,84,101,102,104]. CH_2 angular deformation mode at $1331\text{--}1333\text{ cm}^{-1}$ [101,102], and C-Cl stretching mode at $834\text{--}610\text{ cm}^{-1}$ [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), $\text{CH}_2\text{--Cl}$	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- CaCO_3 particle, by ultrasonic mixing, the results of tensile stress is shown in Fig.2.

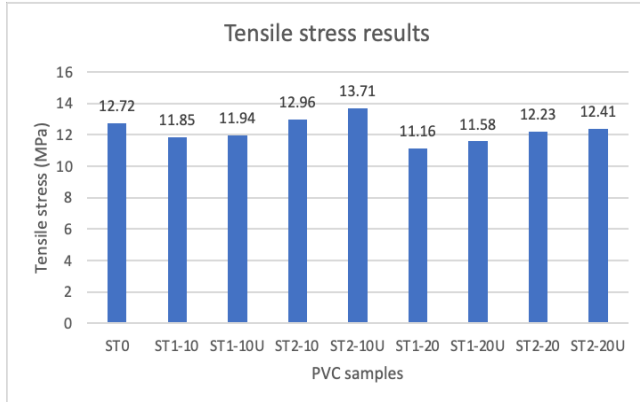


FIGURE 2. Tensile stress results

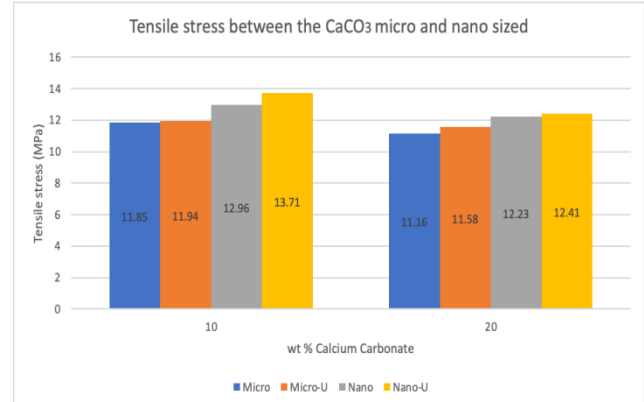


FIGURE 3. Comparing tensile strength between the CaCO_3 micro and nano sized particles

The reason behind increasing the tensile strength in the nano particle of CaCO_3 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 CaCO_3 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 CaCO_3 is higher than micro sized particle of CaCO_3 .

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 CaCO_3 . Elongation at break initially increases and then decreases quickly when the quantity of CaCO_3 amount is greater than 10 wt % as shown in Fig.5. Comparing the PVC samples contain micro size of CaCO_3 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 CaCO_3 have higher elongation than micro.

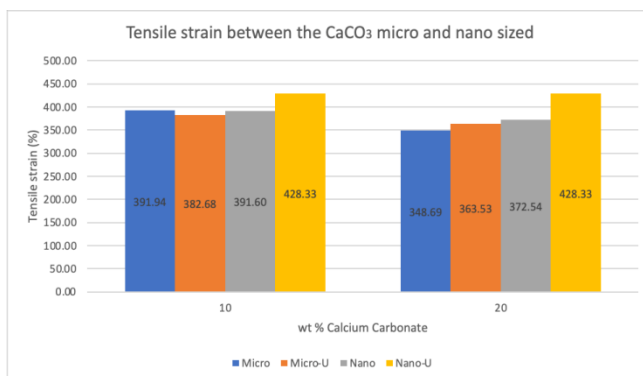


FIGURE 4. Tensile strain results

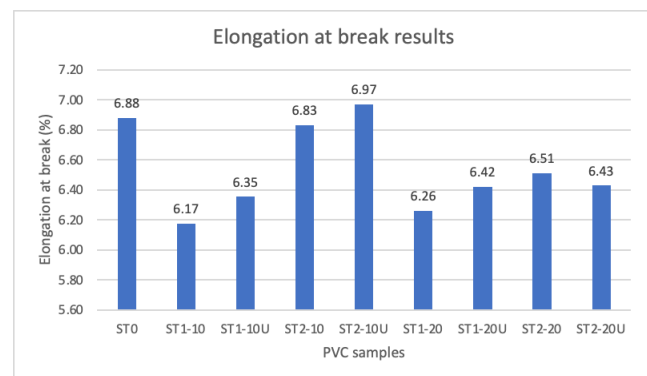


FIGURE 5. Elongation at break results

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.

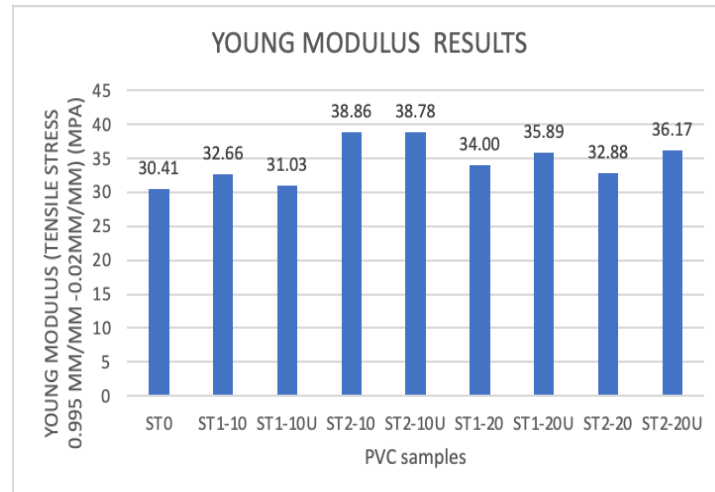


FIGURE 6: Young modulus Results

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.

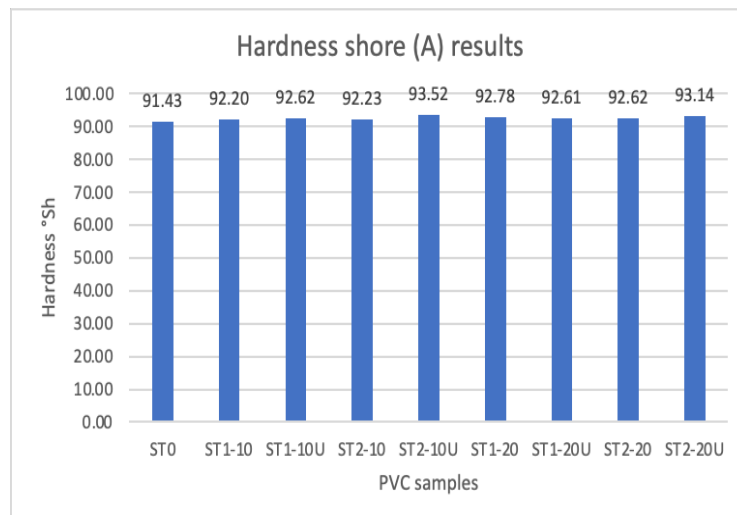


FIGURE 7: Hardness (shore A) results

3.4 TSDC Electrical Test Results and Discussion

The smallest electrical resistance: (ST1-10U) which is (0.9 MΩ), highest electrical resistance: (ST2-20U) which is (6.1 MΩ). The maximum volume resistivity : (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 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.

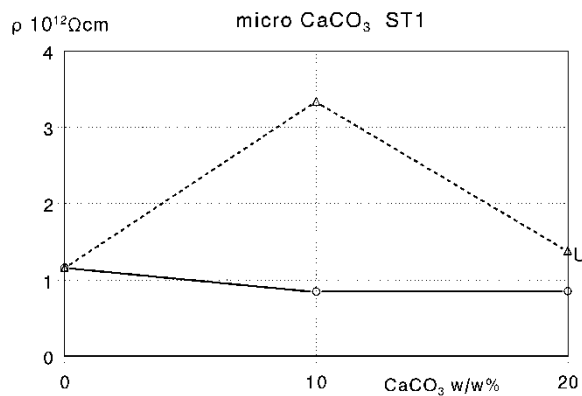


FIGURE 8. Result of electrical resistance and specific volume resistivity of micro sized CaCO_3 filled in PVC

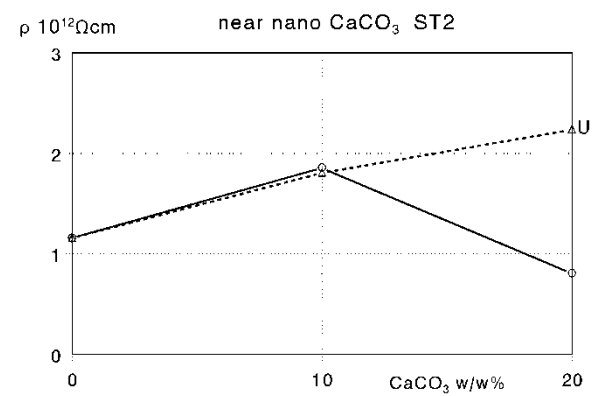


FIGURE 9. Result of electrical resistance and specific volume resistivity of nano sized CaCO_3 filled in PVC

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 CaCO_3 as showed in Fig. 10 and Fig.11.

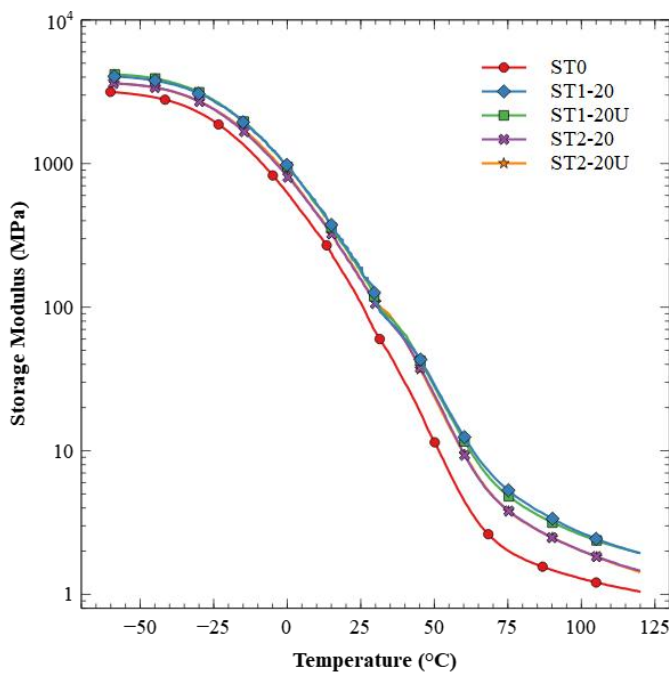


FIGURE 10. Dynamic storage modulus results for PVC samples

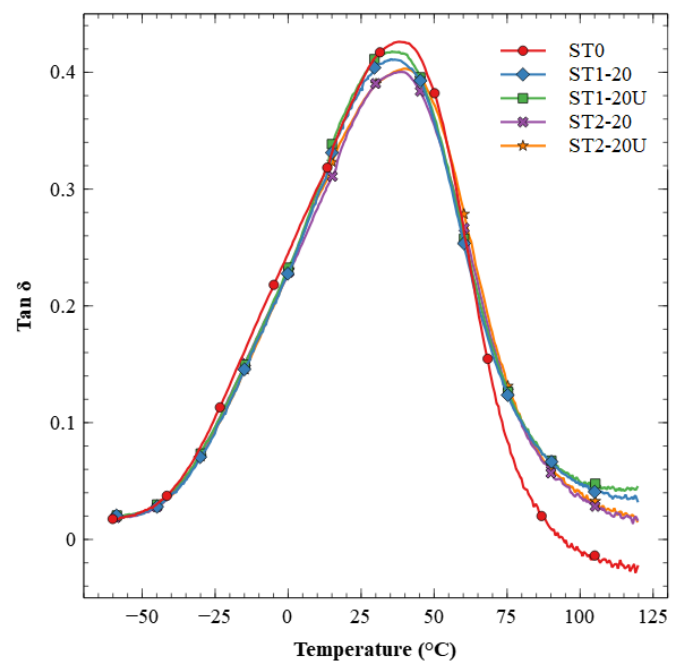


FIGURE 11. Loss factor ($\tan \delta$) results for PVC samples

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 CaCO_3 [78,91]. Loss factor ($\tan \delta$) 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- CaCO_3 , but lower than nano- CaCO_3 samples.

TABLE 3
GLASS TRANSITION TEMPERATURE OF PVC SAMPLES

PVC samples	Glass transition temperature (T_g) (°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
- [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
- [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.; Milanese, 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., & Ajji, 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–FTIR–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., & Rafei, 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] Krzewski, 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-Kalishman, 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(20020101)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-Kalishman, 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(20020101)12:13.0.co;2-c
- [70] Patil, C. B., Kapadi, U. R., Hundiware, 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., Haian, 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: <https://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 chloride (PVC)/sepiolite composite*, **Sakarya University Journal of Science**, vol. 23(4), pp.633-640. DOI:10.16984/aufenbilder.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 Ehtylenediamine: 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.



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