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https://doi.org/10.14233/ajchem.2018.21388 INTRODUCTION Nanocomposites are solid structures with dimensions of nanometre scale and can also be porous, colloid, gel and copolymer. In general, the nanocomposite material demonstrates different mechanical, electrical, optical, electrochemical, catalytic and structural properties from each component [1]. Based on their matrix materials, nanocomposites can be classified into three different groups, namely ceramic matrix nanocomposites, metal matrix nanocomposites and polymer matrix nanocomposites [2]. Polymer matrix nanocomposites, the system consisting of a polymer or copolymer having nanoparticles or nanofillers dispersed in the polymer matrix, have attracted researchers' attention nowadays due to their advantages in physical properties, such as mechanical and thermal properties over the polymeric system [3]. Currently, the most natural material that is widely used as a nano-sized filler for polymer nanocomposites is bentonite. Natural bentonite processing itself can be conducted by several methods, such as co-precipitation method and ball milling method [4,5]. A nano bentonite-making study using ball mill Fabrication of Poly(vinyl alcohol)/Bentonite Nanocomposites Using Sol-Gel Method M. SIRAIT1,*, S. GEA2, N. SIREGAR1, N. BUKIT1, E.M. GINTING1 and R.F. ZEGA1 1Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Negeri Medan, Jl. Willem Iskandar Pasar V Medan Estate, Medan 20221, Indonesia 2Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Sumatera Utara, Jl. Bioteknologi No. 1, Medan 20155, Indonesia *Corresponding author: E-mail: maksir@unimed.ac.id Received: 14 April 2018; Accepted: 2 July 2018; Published online: 31 August 2018; AJC-19045 The aim of this study was to find the characteristics of mechanical, morphological and thermal properties of poly(vinyl alcohol) (PVA)/ bentonite nanocomposites. The method used in making nanocomposites is sol-gel method. The natural bentonite (size of 35.26 nm) obtained from North Sumatra, Indonesia, was added to the solution of poly(vinyl alcohol) with the quantity varied, namely 0, 2, 4, 6, 8 wt %. The nanocomposites yielded was then characterized by using tensile test, scanning electron microscopy and differential scanning calorimetry. The results showed that the maximum weight of added bentonite, which improved the mechanical properties and produced more homogenous surface, was 6 wt %, whereas the variation of 2, 4 and 8 wt % of bentonite resulted in a decreased elasticity modulus. However, thermal properties is preferable to improve in the 0 wt % of bentonite, Keywords: Bentonite, Poly(vinyl alcohol), Sol-gel method, Nanocomposites. Asian Journal of Chemistry; Vol. 30, No. 10 (2018), 2210-2214 This is an open access journal, and articles are distributed under the terms of the Creative Commons Attribution-NonCommercial 4.0 International (CC BY-NC 4.0) License, which allows others to copy and redistribute the material in any medium or format, remix, transform, and build upon the material, as long as appropriate credit is given and the new creations are licensed under the identical terms. and co-precipitation method was obtained with size 35.26 nm [6]. The use of bentonite as reinforcement material for polymer nanocomposites is expected to give preferable modulus, strength and heat resistance. There has been considerable research on the use of bentonite as a filler in polymeric materials due to the high biocompatibility and good gel-forming capability of the polymer [7]. Bukit et al. [8] found that the application of bentonite as nanofillers for high density polyethylene (HDPE) polymeric system could enhance the mechanical properties of the system with the best performance at 2-6 wt % of bentonite. Similarly, Ginting et al. [9] discovered that the addition of Na-bentonite filler and carbon black could influence the rubber. Related to that, the

utilization of bentonite to other polymeric systems needs to be conducted in order to develop the study about polymer nanocomposites. One of the polymer matrix which has huge application is poly(vinyl alcohol). Besides having hydrophilicity and biocompatibility properties, it also has good thermal stability and is considered as nontoxic material. Because of those properties, PVA has a wide application, such as in home textiles, coating materials and industrial materials [10]. In order to enhance its properties, namely mechanical and thermal properties, some reinforcement materials are applied as the fillers. Turhan et al. [11] attempted to fabricate nanocomposites of PVA/modified bentonite, found that there was an improvement in the thermal stabilities over the pure PVA. The method used was the solution intercalation method. Also, FerrándezRives et al. [12] conducted the research about PVA/bentonite by using electrospinning, they discovered that the nanocomposites gave better properties for the biomedicine and biotechnology application. In addition, Sang et al. [13] prepared composite solid alkaline polymer electrolyte PVA-bentoniteKOH-H2O using solution-casting method and found that the interpolation of bentonite to PVA matrix could increase the KOH content in PVA matrix, thus it improved the conductivity of the system particularly at higher water content. Based on above explanation, this study aims to investigate the characteristics of mechanical properties, morphology and thermal nanoscale composites on the addition of nano mixtures of natural bentonite particles to the PVA polymer using solgel method. Sol-gel method is one of the fastest growing chemical fields and is the process of forming inorganic compounds via chemical reactions at low temperature solution, while in the process; there is a phase change from the colloidal suspension (sol) to the liquid phase (gel) [14]. It was employed due to its procedure that is based mainly in water soluble polymers, such as PVA. Therefore, the fabrication of nanocomposite PVA/bentonite using sol-gel method should give another perspective of the study about nanocomposites. EXPERIMENTAL The materials used in this study were synthetic bentonite which is obtained from North Tapanuli Pahae District, North Sumatra, Indonesia with size of 35.26 nm [6,15], aquabidest and poly(vinyl alcohol) (PVA) pro-analyst purchased from Sigma-Aldrich. The characterization was conducted using several equipments, namely universal testing machine model UCT-5T by Orientec Corporation Ltd. to test mechanical properties, scanning electron microscopy (SEM) EVO MA 10 Zeiss to discover the surface morphology and differential scanning calorimetry (DSC) (Mettler Toledo type 821) to test the thermal properties. The synthesis of PVA/bentonite nanocomposite was performed by sol gel method. As much as 24 g of PVA were dissolved into 200 mL of distilled water and stirred at 550 rpm and at 80 'C. After the PVA was completely dissolved, bentonite nanoparticles were added into the solution with the variations of 0, 2, 4, 6 and 8 % for 7 h at 80 'C and stirred 550 rpm until thickened to gel. After the solution thickened, it was inserted into the glass mold to dry and harden at room temperature. Then the sample was printed using ISO 527-2 type 1A for Universal Testing Machine UCT-5T model. Then the sample was characterized by using tensile and SEM. DSC was applied to determine the melting point of the nanocomposite PVA/bentonite. RESULTSAND DISCUSSION Mechanical properties analysis: The tensile test was performed to evaluate the effect of the addition of bentonite on the mechanical properties of the nanocomposite membrane. Fig. 1 shows the tensile strength of PVA/bentonite nanocomposites with the variations in the quantity of bentonite. Tensile strength testing on PVA/bentonite showed that there was an enhancement when the clay content increased from 0 to 2 %, but this value dropped when 4 % of bentonite obtained. The fluctuation kept occurring until 8 % of clay content fulfilled. The strength of PVA/bentonite nanocomposites had a maximum value of 6 % by weight of bentonite. The tensile strength of the PVA/bentonite nanocomposites was largely caused by the agglomeration of the organoclay particles [16]. This result appears to be associated with a lack of interfacial interactions between clays and polymer matrices. Thus, the large number of defects and agglomerations occurred in the interphase region [17-19]. At the strain value, it was found that for each variation indicated a 100 % increasing as shown in Figs. 2 and 3. This increasing occured because the dispersed nanobentonite in the polymer matrix will lead to an improvement in the mechanical properties of the membrane nanocomposite. It has been reported that the addition of coated filler particles can easily improve the mechanical properties of the polymer [20]. 30 25 20 15 10 5 0 Tensile strength (MPa) 02468 Quantity of bentonite (wt %) Fig. 1. Tensile strength of PVA/bentonite nanocomposites Fig. 4 suggests that the addition of bentonite may increase the flexibility of catalyst in PVA [21]. The sharp increase in the addition of nano-bentonite would have an impact on the value of the Young Modulus. It can be assumed that at the high value of increasing in length will cause a low modulus value [22]. Finally, based on Fig. 5, it can be seen from the modulus of elasticity of the 6 % bentonite composition has a higher elasticity modulus value compared to the pure PVA. This increasing occured because there was a strong interaction between the matrix and silicate layers through the formation of hydrogen bonds due to the strong hydrophilicity of the bentonite edge. However, there was also a decreasing in elasticity modulus value in the variation of 2, 4 and 8 % bentonite. It was due to the deployment of bentonite to the PVA which was uneven [23]. Vol. 30, No. 10 (2018) Fabrication of Poly(vinyl alcohol)/Bentonite Nanocomposites Using Sol-Gel Method 2211 120 100 80 60 40 20 0 Elongation at break (mm) 02468 Quantity of bentonite (wt %) Fig. 2. Elongation at break of PVA/bentonite nanocomposites 450 400 350 300 250 200 150 100 50 0 Strain (%) 02468 Quantity of bentonite (wt %) Fig. 3. Strain of PVA/bentonite nanocomposites Morphology analysis: The gel morphology of each variation of the PVA/bentonite nanocomposites in which the nanobentonite is dispersed on the PVA polymer matrix is shown by Fig. 5. In detail, Fig. 5b, a 2 % PVA/bentonite variation showed a curves and pores on the surface of the gel with a curvature length of 4.122 um, while the pore length was 1.342 um. In variation of 4 %, it contained three pores with different sizes, while for variation of 8 % have many pores. For the variation of 6 %, the surface was observed to be flatter, so that fewer pores with a pore length of 0.5237 um were formed compared to other variations, this was caused by a better bond between the matrix with the filler compared with others. 140 120 100 80 60 40 20 0 Young's modulus (MPa) 02468 Quantity of bentonite (wt %) Fig. 4. Young's modulus of PVA/bentonite nanocomposites The connection between PVA with bentonite can be stable because of the hydrogen bond [24]. The interaction rate between the PVA polymer molecule and bentonite particles depends on the polymer concentration in the dispersion [15]. Bentonite can strengthen a crosslinked PVA nanocomposites membrane with various bentonite concentrations [25]. Thermal properties analysis: The main information gained from DSC analysis regarding thermal properties is the heat involved in thermal event as a function of time and temperature. The heat involved is commonly expressed in term of enthalpy, which is the amount of heat released or adsorbed by the sample during thermal treatment. The negative value of enthalpy means that the sample adsorbs heat, as indicated by the endothermic peak on the DSC thermogram. In addition, DSC analysis provides quantitative information about melting point and phase transition of the sample [26]. As can be seen in Fig. 6, all samples melt at practically the same temperature, but with different enthalpies. From the analysis of DSC in Fig. 6 and Table-1 on the PVA/bentonite particles (0-8) wt % visible the melting point did not increase significantly with the increase of bentonite at 0 % of melting point value 223.51 'C with enthalpy of 27.10 J/g, however at the 2-6 % composition there is an increase. These results are in accordance with the references (210-230) 'C for full hydrolysis of PVA. TABLE-1 RESULTS OF THERMAL TESTING OF DSC NANOCOMPOSITE PVA/BENTONITE Temperature ('C) Bentonite (wt %) Onset Melting Endset Entalphy (ΔH, J/g) Heat (ΔQ, mJ) 0 209.05 223.51 228.72 27.10 111.12 2 198.41 219.98 224.89 46.83 192.00 4 208.06 222.09 227.24 62.65 256.87 6 208.15 221.95 227.48 49.65 203.56 8 207.27 221.48 227.37 22.71 93.09 2212 Sirait et al. Asian J. Chem. Fig. 5. SEM photograph of PVA/bentonite nanocomposites: (a) pristine PVA, (b) PVA/bentonite with 2 wt % bentonite, (c) PVA/bentonite with 4 wt % bentonite, (d) PVA bentonite with 6 wt % bentonite, (e) PVA/bentonite with 8 wt % bentonite 27 26 25 24 23 22 21 20 19 18 Heat flow end to up (mW) 50 100 150 200 250 300 350 Temperature ('C) 0 wt % Bentonite 2 wt % Bentonite 4 wt % Bentonite 6 wt %

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Bentonite 8 wt % Bentonite Fig. 6. Overlay thermogram of differential scaning calorimetry (DSC) from
PVA/bentonite with bentonite varied 0-8 wt % Conclusion Based on the results of mechanical tests of
PVA/bentonite nanocomposites, the composition of bentonite (94:6) % gained an average value of the
largest Young's modulus of 114.71 MPa. This mixture also obtained a uniform and homogenous
morphologhy. Thermal properties is preferable to occur in the composition of 100:0 %, obtained the
melting temperature of 223.51 'C with enthalpy of 27.10 J/g and the heat of 111.12 mJ.
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[11:22:51 AM] YahFound 1% matches at: http://file.scirp.org/pdf/MSA20110700013_98524023.pdf
[11:22:51 AM] The page wasn't loaded from the query #104-1 (30090 ms., timeout exceeded 30000 ms.):
https://www.science.gov/topicpages/b/bentonite+swelling+pressure
                 The page wasn't loaded from the query #104-3 (30090 ms., timeout exceeded 30000 ms.):
[11:22:51 AM]
https://worldwidescience.org/topicpages/s/soil-bentonite+slurry+walls.html
[11:23:01 AM] The page wasn't loaded from the query #111-3 (30611 ms., timeout exceeded 30000 ms.):
http://iopscience.iop.org/article/10.1088/1742-6596/340/1/012090/pdf
[11:23:10 AM]
                 The page wasn't loaded from the query #115-2 (30033 ms., timeout exceeded 30000 ms.):
https://scholarsmine.mst.edu/cgi/viewcontent.cgi?article=1582&context=isccss
[11:23:15 AM] YahFound 1% matches at: https://www.bing.com/aclick?ld=d3p3xOdxr-
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GGI40I2NicrUMdF9qiClshRa2E2IZ86ivpb-0yEq8YO5rtt658jP4bCgfWPco7JEyg6n9cob-rC5VZ8_-
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0dsc%20analysis%20regarding%20thermal%20properties%20heat&url=https://www.mt.com/us/en/home/pr
oducts/Laboratory Analytics_Browse/TA_Family_Browse/DSC.html?cmp=sea_06040127&SE=BING&Ca
mpaign=MT ANA-TA EN US&Adgroup=DSC+-
+Measurement&bookedkeyword=%2Bdsc%20%2Bthermal%20%2Banalysis&matchtype=b&adtext=78683
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not_available&network=o&kclid=_kenshoo_clickid_&rlid=1c20101bfe691856bc704be738f910b7
[11:23:16 AM] Go Found 1% matches at: https://www.hindawi.com/journals/jnm/2015/291248/
[11:23:18 AM] The page wasn't loaded from the query #118-1 (30022 ms., timeout exceeded 30000 ms.):
http://sites.ieee.org/ocs-cpmt/files/2013/06/130623 IEEE kyocera chemical.pdf
                 The page wasn't loaded from the query #104-3 (30039 ms., timeout exceeded 30000 ms.):
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https://worldwidescience.org/topicpages/s/soil-bentonite+slurry+walls.html(Saved copy)

http://article.sciencepublishinggroup.com/pdf/10.11648.j.ajpc.20140301.12.pdf

[11:23:31 AM] Bi Found 1% matches at:

[11:23:28 AM] Go Found 1% matches at: https://www.ncbi.nlm.nih.gov/pmc/articles/PMC3053520/

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[11:23:31 AM] Go Found 1% matches at: https://www.ncbi.nlm.nih.gov/pmc/articles/PMC4204778/
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https://courses.helsinki.fi/sites/default/files/course-material/4537964/Lect 5 DTA-DSC engl.pdf
                 The page wasn't loaded from the query #133-3 (30057 ms., timeout exceeded 30000 ms.):
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http://data-gov.tw.rpi.edu/raw/1560/data-1560-00053.rdf
[11:23:52 AM] Go Found 4% matches at: https://docplayer.net/20984012-Mechanical-and-thermal-
properties-of-polypropylene-reinforced-by-calcined-and-uncalcined-zeolite.html
[11:23:55 AM] YahFound 1% matches at: https://www.avs.org/AVS/files/61/61088ed4-a189-469b-9393-
fba7570c8e76.pdf
[11:23:56 AM] Go Found 1% matches at: https://www.ncbi.nlm.nih.gov/pmc/articles/PMC5632318/
[11:24:04 AM] The page wasn't loaded from the query #142-1 (30055 ms., timeout exceeded 30000 ms.):
https://www.facebook.com/notes/farshad-fym/free-proxy-ip-port/200599046617892
[11:24:06 AM] YahFound 1% matches at: https://www.hindawi.com/journals/amse/2015/576080/
[11:24:08 AM] The page wasn't loaded from the query #140-3 (30057 ms., timeout exceeded 30000 ms.):
https://books.google.co.id/books?id=4fK6L6RxJkYC&pg=RA1-PA77&lpg=RA1-
PA77&dq=(+06+222+09+227+24+)+|+(+62+65+256+87+6+)&source=bl&ots=Ts6qDwT0Dm&sig=PPfQD
TMy0Qbg5czVmxcCujRKocw&hl=ru&sa=X&ved=2ahUKEwi8sI363PPeAhVMX30KHU0SBngO6AEwA
noECAcQAQ
[11:24:12 AM] YahFound 1% matches at: https://benthamopen.com/EPUB/BMS-TOPSYJ-2018-48
[11:24:18 AM] The page wasn't loaded from the query #148-1 (30036 ms., timeout exceeded 30000 ms.):
https://www2.gov.bc.ca/assets/gov/farming-natural-resources-and-industry/electricity-alternative-
energy/04_majorowicz_sed_basins.pdf
[11:24:18 AM] The page wasn't loaded from the query #148-3 (30040 ms., timeout exceeded 30000 ms.):
https://www.sintef.no/globalassets/project/trondheim_gts/presentasjoner/mechanical-integrity-of-the-pfhe-in-
Ing-liquefaction-process.pdf
[11:24:18 AM] The page wasn't loaded from the query #147-2 (30075 ms., timeout exceeded 30000 ms.):
http://www.hitachi-dentetsu.co.jp/bus/bus_osirase/20180702_jikokuhyo_BRT.pdf
                 The page wasn't loaded from the query #151-3 (30071 ms., timeout exceeded 30000 ms.):
http://chemistry-reference.com/Standard Thermodynamic Values.pdf
[11:24:32 AM] YahFound 1% matches at:
http://www.deswater.com/DWT references/vol 60 references/60 2017 261.html
[11:24:32 AM] Bi Found 1% matches at: https://link.springer.com/article/10.1023/A:1010992820048
[11:24:34 AM] Go Found 1% matches at: https://www.ncbi.nlm.nih.gov/pmc/articles/PMC5908830/
[11:24:41 AM] Bi Found 1% matches at: https://link.springer.com/article/10.1007/BF00455608
[11:24:47 AM] Go Found 1% matches at: https://www.neliti.com/id/universitas-negeri-
medan?per_page=50&page=5
[11:24:52 AM] Bi Found 1% matches at: https://www.ncbi.nlm,nih.gov/pmc/articles/PMC4954872/
[11:24:52 AM] Bi Found 1% matches at: https://link.springer.com/article/10.1007/s00500-018-3635-6
[11:24:55 AM] The page wasn't loaded from the query #166-3 (30025 ms., timeout exceeded 30000 ms.):
http://iopscience.iop.org/article/10.3847/2041-8213/aab889/meta
[11:25:00 AM] Go Found 1% matches at: https://www.hindawi.com/journals/jchem/2015/385741/ref/
[11:25:00 AM] Go Found 1% matches at:
http://www.sciencepublishinggroup.com/journal/paperinfo.aspx?journalid=227&doi=10.11648/j.ajac.s.2015
030301.18
[11:25:02 AM] Go Found 1% matches at: https://www.hindawi.com/journals/jnm/2014/967492/
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[11:25:02 AM] YahFound 1% matches at:

http://scholar.google.com/citations?user=9q88YtAAAAAJ&hl=en

[11:25:03 AM] YahFound 1% matches at:

http://scholar.google.com/citations?user=uMIqrq0AAAAJ&hl=en

[11:25:05 AM] Bi Found 1% matches at: https://link.springer.com/journal/11581/21/2/page/2

[11:25:08 AM] Bi Found 1% matches at: https://link.springer.com/article/10.1007/s11581-014-1193-1

[11:25:09 AM] Go Found 12% matches at:

http://www.asianjournalofchemistry.co.in/Journal/ViewArticle.aspx?ArticleID=30_10_10&fileType=XML

[11:25:11 AM] YahFound 1% matches at: https://link.springer.com/article/10.1023/A:1020818414927

[11:25:16 AM] YahFound 1% matches at: https://www.cambridge.org/core/journals/journal-of-materials-research/article/effect-of-thermally-stable-oligomerically-modified-clay-on-the-properties-of-aramidbased-nanocomposite-materials/5505FDBD251F99D14D5FEE04A50FE59C

[11:25:17 AM] YahFound 1% matches at: http://pubs.rsc.org/en/content/articlehtml/2015/ra/c5ra22620a

[11:25:19 AM] Go Found 1% matches at: https://link.springer.com/article/10.1007/s13233-013-1170-y

[11:25:22 AM] Go Found 1% matches at:

http://www.koreascience.or.kr/article/ArticleFullRecord.jsp?cn=GBJHCY_2013_v37n2_225

[11:25:22 AM] Go Found 1% matches at:

http://www.koreascience.or.kr/article/ArticleFullRecord.jsp?cn=GBJHCY_2013_v37n5_579

[11:25:30 AM] Bi Found 1% matches at: https://link.springer.com/chapter/10.1007/978-90-481-2567-8_5

[11:25:39 AM] Go Found 1% matches at: https://docplayer.net/31296664-Sains-materi-indonesia.html

[11:25:45 AM] Check type: *Deep*

[11:25:45 AM] The originality of the text is 80%[©]

