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Polylactide and its Composites on Various Scales of Hardness

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ABSTRACT

Polylactide (PLA) has become a widely applied material. Its hardness property has, however, not been a subject of intense study. This study attempts to examine the hardness values of Polylactide and its composites on ten hardness scales. Polylactide composites were developed using three reinforcements (i.e., chitosan, chitin, and titanium powders). The compositing method was the melt-blending technique. Vickers microindentation test was carried out on all the developed samples. The experimental values obtained were related to nine (9) other scales of hardness via an online reference interface. Results showed that the Brinell and Rockwell hardness scales agreed, to a large extent, with the experimental values from several studies. Hence, this work can serve as a reference material on the Brinell and Rockwell hardness values of the unreinforced and reinforced composites considered in this study. The developed materials were also represented on the Mohs scale of hardness with unreinforced PLA having the least value of hardness which corresponds to the value of gypsum on the Mohs scale while the PLA reinforced with 8.33 weight (wt.) % of titanium powder has the highest value of hardness corresponding to the value of a material in-between calcite and fluorite. The hardness values obtained on Shore scleroscope could not agree with the experimental values from various studies. Succinctly, the three particulate fillers increased the hardness properties of PLA. The results of this study would go a long way in helping industrialists and researchers in the correct applications of PLA and its composites.

Keywords: Annealed polylactic acid; biodegradable polymer; Knoop scale; polylactide composites; Rockwell hardness scale

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INTRODUCTION

Polylactide (PLA) or polylactic acid (PLA) has been well researched in recent times because of some of its preferred properties compared to the petroleum-based polymers (Adeosun et al., 2016). Contingent upon its desirable characteristics, it has grown to become a widely applied biopolymer (Abreu et al., 2017; Deepthi et al., 2016). However, there is a need to improve on some of its drawbacks in order to give it a wider area of applications (Abreu et al., 2017). Researchers, in recent years, have worked to improve on its hydrophobicity (Aworinde et al., 2020a; Hendrick & Frey, 2014; Kurzina et al., 2020; Qi et al., 2019), modulus of elasticity (Adeosun et al., 2016; Aworinde et al., 2020b; Wang et al., 2016), brittleness (Kunwar et al., 2012; Liu & Zhang, 2011; Sennan & Pumchusak, 2014; Song et al., 2014), and so on. Notwithstanding the efforts that have been deployed to widen the areas of application of PLA through properties modification, there appears to be a need for more intense characterisation. The hardness properties of PLA, for instance, has not been seen as an advantage. Improving on it has, therefore, not been the focus of researchers. Yet studies have shown that improved hardness is an empirical result of reinforcing PLA with particulate fillers (Aworinde et al., 2020c; Aworinde et al., 2019a; Feng et al., 2016), and several particulate-reinforced PLA composites have been developed for both the soft (Adeosun et al., 2016; Deepthi et al., 2016; Gbenebor et al., 2018) and the hard (Aworinde et al., 2020c; Wang et al., 2016) tissues applications. Aside from the use of particulate reinforcements, developing PLA composites requires the use of certain polymer composites processing techniques (Aworinde et al., 2018; Aworinde et al., 2019b; Deepthi et al., 2016), which often impact the hardness properties of PLA significantly (Vian & Denton, 2018).

The microindentation properties of a material can be examined on several available scales of hardness. The understanding of the values of the hardness of a material such as PLA on various scales would enhance the right application of the material. The hardness test is one of the most performed mechanical tests because of its usefulness and versatility and Vickers hardness has been reported to be one of the oldest methods of determining the hardness properties of materials because of its wide hardness scale (Moore & Booth, 2015). From Vickers (HV) hardness test, for instance, a number of other helpful parameters can be measured to give a reasonable prediction of other mechanical properties such as fracture toughness (Bergner et al., 2007; Moradkhani et al., 2013) as described in our previous study (Aworinde, et al., 2020c). In addition to the Vickers hardness test, a few other available hardness tests include (though not limited to) Brinell hardness test (HB), Knoop hardness test (HK), Janka hardness test, Meyer hardness test, Rockwell hardness test (HR), Shore durometer hardness test and Barcol hardness test are a few of the available hardness tests.

Apart from the fact that a wide knowledge of material's hardness predisposes it to a wide area of applications, material for implants are expected to possess, among other critical mechanical properties, some degrees of wear resistance (Chandrasekaran, 2010).

In addition, recent studies are focusing on the abrasive/wear resistance of PLA for diverse applications (Eutionnat-Diffo et al., 2020; Mohd et al., 2019). Interestingly, research has shown that there exists a relationship between material hardness and its wear resistance (Faria et al., 2007; Luyckx & Love, 2004; Mezlini et al., 2009). The premise is that the harder the material, the greater the wear resistance. In the light of several possibilities ahead of PLA, this work assays to present its hardness values and those of its composites on various scales of hardness. This material of the future (i.e., PLA), as it is often called, has the propensity to replace many materials in the nearest future (Anderson & Shenkar, 2021; Cooper, 2013). It, therefore, becomes necessary to examine its hardness on various scales of hardness, because different materials possess widely varying hardnesses and require different scales for testing.

MATERIALS AND METHODS

The materials used in this study include PLA, chitosan, chitin, and titanium powder. Our earlier work (Aworinde et al., 2020a; Aworinde et al., 2020c) details the properties of the materials and how they were obtained. Melt-blending technique was used to produce three PLA composites with chitosan, chitin, and titanium (Ti-6Al-2Sn-2Mo-2Cr-0.25Si) powders as reinforcements. The three composites produced are designated as PLA/Ch (i.e., chitosan reinforced PLA), PLA/Ct (i.e., chitin reinforced PLA) and PLA/Ti (i.e., titanium-reinforced PLA). The maximum particle size of chitosan and chitin was 75 µm while the average particle size of titanium powder was 67.5 µm.

Chitosan and chitin are organic fillers, while titanium is an inorganic filler. The choice of these fillers thus helped to compare the effects of organic and inorganic reinforcements on the hardness value of PLA. Our previous studies explain, in detail, the composite fabrication method employed in the development of the samples (Aworinde et al., 2020a; Aworinde et al., 2020c), matrix-fillers formulation and the details of Vickers microhardness characterisation (Aworinde et al., 2020c). The microhardness indentation dwell times were chosen in accordance to ASTM standard (ASTM-E384, 2017).

In order to obtain the approximate equivalent hardness values of Polylactide and Polylactide composites on other hardness scales, the experimental values obtained from the Vickers hardness test was supplied to the interface of an online engineering reference (Efunda, 2020). Approximate values of hardness on Brinell hardness scale with 3000 kgf indentation load, Brinell hardness scale with 500 kgf indentation load, Knoop scale, Mohs scale, Rockwell B-scale, Rockwell Superficial 15T Scale, Rockwell Superficial 30T Scale, Rockwell Superficial 45T Scale and Shore Scleroscope were obtained.

RESULTS AND DISCUSSION

It was observed throughout the results obtained from this study that chitin improved the hardness of PLA better than chitosan. This is undoubtedly connected with the stronger structural property of Chitin over Chitosan, because of the difference in their degrees of de-acetylation. Also, an investigation between the hardness property of PLA/Ct and PLA/Ti showed superior hardness values of the later over PLA/Ct. This was in order since metallic powders are expected to produce polymer composites with higher mechanical properties than would a natural polymer filler (Premalal et al., 2002). However, further addition of titanium beyond 8.33 wt. % showed a drastic reduction in hardness, making PLA/Ti to be 27.66 % and 1.51% less hard than PLA/Ct and PLA/Ch, respectively. Traditionally, this is not expected since titanium is far structurally stronger than Chitin and Chitosan as observed in other composites with less concentration. These exceptions may be due to poor dispersion of the reinforcement in the matrix as Ti increased, thereby leading to poor miscibility: a condition peculiar to polymer-metal composites than polymer-polymer composites (Pozuelo et al., 2017; Souza et al., 2019).

Table 1 presents the Vickers hardness values of PLA and its composites obtained from the experiment. Table 2-10 give the approximate figures of hardness values on several hardness scales, namely: Brinell hardness scale with 3000 kgf indentation load (Table 2), Brinell hardness scale with 500 kgf indentation load (Table 3), Knoop scale (Table 4), Mohs scale (Table 5), Rockwell B-scale (Table 6), Rockwell Superficial 15T Scale (Table 7), Rockwell Superficial 30T Scale (Table 8), Rockwell Superficial 45T Scale (Table 9) and Shore Scleroscope (Table 10). The equivalent values on other hardness scales which could not be obtained from the online reference have been indicated by << (i.e., below the minimum obtainable value) and >> (i.e., above the maximum obtainable value).

The approximate value of PLA/Ti at 8.33 wt. % of titanium filler on the Brinell 10 mm Standard 3000 kgf Scale (i.e., 254 BRH) relates well with the value obtained from experiments on a Brinell scale of hardness (i.e., 265 BRH) when PLA was reinforced with 15 wt. % of carbon (Mohan et al., 2019). Although several studies did not state, in particular, the type of Rockwell scale of hardness used, the values of Rockwell B-scale, Rockwell Superficial 15T Scale and Rockwell Superficial 30T Scale obtained in this work form a close range with the experimental values obtained for annealed PLA (Farah et al., 2016). This shows that the values obtained in this study approximate, with minimal error, the hardness values of PLA and its composites on the various scale of hardness. The predicted values of hardness on the shore scleroscope scale seem not to be in agreement with the experimental values reported in various studies on Shore D scale (Byrne et al., 2009; Valerga et al., 2020). The values on Shore D scale from various studies are generally higher than the approximate equivalent values obtained from the online reference on Shore scleroscope scale. This may be attributed to the fact that there is no good correlation between the Vickers hardness scale and shore scleroscope scale.

The values of PLA and its composites on the Mohs scale imply that an unreinforced, heat-treated PLA was as hard as gypsum and can, therefore, be scratched with the fingernail (King, 2020). PLA composites, however, have higher hardness values. PLA/Ch, for instance, has a hardness value between gypsum (i.e. 2) and calcite (i.e. 3) while PLA/Ct with 8.33 wt. % has the same hardness value as calcite. On the other hand, PLA/Ti with 8.33 wt. % reinforcement has a value that is characteristic of materials with the hardness value between calcite and fluorite. Studies are very scanty on the hardness values of PLA and its composites on the Mohs scale of hardness as well as on the Knoop scale.

Table 1
Vickers hardness scale

Filler (wt. %)	PLA/Ch	PLA/Ct	PLA/Ti
0.00	72.8	72.8	72.8
1.04	76.1	150.6	151.8
2.08	90.2	130.2	161.8
4.17	107.2	148.1	180.5
8.33	148.4	203.9	268.1
16.67	167.7	166.6	165.2

Table 2
Brinell 10 mm standard 3000 kgf scale

Filler (wt. %)	PLA/Ch	PLA/Ct	PLA/Ti
0.00	69	69	69
1.04	72	144	145
2.08	86	124	154
4.17	102	141	172
8.33	141	194	254
16.67	160	159	157

Table 3
Brinell 10 mm standard 500 kgf scale

Filler (wt. %)	PLA/Ch	PLA/Ct	PLA/Ti
0.00	<<	<<	<<
1.04	<<	131	132
2.08	81	114	140
4.17	95	129	154
8.33	129	170	>>
16.67	144	144	142

Table 4
Knoop scale

Filler (wt. %)	PLA/Ch	PLA/Ct	PLA/Ti
0.00	90	90	90
1.04	93	163	164
2.08	106	143	174
4.17	122	160	192
8.33	160	214	278
16.67	179	178	177

Table 5
Mohs scale

Filler (wt. %)	PLA/Ch	PLA/Ct	PLA/Ti
0.00	2.0	2.0	2.0
1.04	2.0	2.5	2.5
2.08	2.5	2.5	2.5
4.17	2.5	2.5	3.0
8.33	2.5	3.0	3.5
16.67	2.5	2.5	2.5

Table 6
Rockwell B-scale

Filler (wt. %)	PLA/Ch	PLA/Ct	PLA/Ti
0.00	<<	<<	<<
1.04	32	79	79
2.08	47	72	82
4.17	60	78	87
8.33	78	92	95
16.67	84	84	83

Table 7
Rockwell superficial 15T scale

Filler (wt. %)	PLA/Ch	PLA/Ct	PLA/Ti
0.00	<<	<<	<<
1.04	<<	87	87
2.08	76	84	88
4.17	80	86	90
8.33	86	91	94
16.67	88	88	88

Table 8
Rockwell superficial 30T scale

Filler (wt. %)	PLA/Ch	PLA/Ct	PLA/Ti
0.00	<<	<<	<<
1.04	<<	70	70
2.08	48	65	72
4.17	56	69	76
8.33	69	79	>>
16.67	74	73	73

Table 9
Rockwell superficial 45T scale

Filler (wt. %)	PLA/Ch	PLA/Ct	PLA/Ti
0.00	<<	<<	<<
1.04	<<	53	54
2.08	20	45	57
4.17	33	52	62
8.33	52	67	>>
16.67	58	58	58

Table 10
Shore scleroscope

Filler (wt. %)	PLA/Ch	PLA/Ct	PLA/Ti
0.00	<<	<<	<<
1.04	<<	22	22
2.08	<<	19	24
4.17	16	22	26
8.33	22	29	38
16.67	25	24	24

CONCLUSION

A holistic view of the consequence of particulate reinforcement of PLA, as it affects its hardness property, has been taken in this study. Unreinforced PLA and its three composites prepared by melt-blending technique were subjected to Vickers microhardness test. The results were examined on nine (9) other scales of hardness via an online reference interface and the outcome compared with the experimental results from other studies. The results form a database for comparing the hardness properties of PLA and its composites with the hardness values of various materials (e.g. petroleum-based plastics) for the possibility of getting a viable replacement. Also, it was observed that:

i. in tandem with other research results, the particulate reinforcements used increased the hardness values of the matrix (i.e., PLA)
ii. PLA composites as hard as calcite and fluorite can be produced using the simple melt-blending technique.
iii. the Vickers hardness scale bears a good correlation with several other scales of hardness such as Brinell and Rockwell, but not with the shore scleroscope scale of hardness.
iv. the availability of data as presented in this study would minimise the number of experimental runs and hence reduce material and time wastages in research and development.

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