

ASEAN JOURNAL ON SCIENCE & TECHNOLOGY FOR DEVELOPMENT

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Fabrication, Rheology and Antioxidant Activity of Palm Esters-based Emulsions Loaded with Tocotrienol

S. H. NG^{1*}, B. MAHIRAN² AND I. ZAHARIAH³

Palm oil esters are wax esters derived from palm oil and oleyl alcohol. Palm oil esters have many applications due to their luxurious moisturizing properties, non-greasy and blend easily with fragrances and colours when applied on skin surface. The aim of this research was to fabricate palm esters-based emulsions cream for topical delivery, characterise the rheological properties and *in vitro* antioxidant activity of the palm esters-based emulsions system.

Emulsions containing palm oil esters loaded with tocotrienol were obtained in two stages, with propagation of rotor-stator at 6000 r.p.m. for 5 min and further emulsified using an ultrasound at various acoustic amplitudes for another 5 min. A stress/rate controlled Kinexus Rheometer with a temperature controller was used to measure the rheological properties of the emulsion. Rheology measurements were performed at 25.0° C \pm 0.1°C with 4°/40 mm cone and plate geometry. The *in vitro* antioxidant activity was investigated using UV-Vis spectrophotometer.

The yield stress of the emulsions increased with increasing acoustic amplitudes. The viscoelasticity of the emulsions were enhanced by the increase in the oil and surfactant concentrations. The emulsions with higher oil phase concentration [30% (w/w)] showed greater elasticity which implied strong dynamic rigidity of the emulsions. The cohesive energy increased significantly with surfactant concentration especially for the emulsions with 30% (w/w) oil phase concentration. The palm oil esters emulsions containing tocotrienol gave higher Trolox equivalent antioxidant capacity values which implied higher antioxidant capability.

The tocotrienol in emulsion with 30% (w/w) dispersed phase showed that they were the most stable with longest shelf life and exhibited greater inhibitory effects on the ABTS⁺⁺.

Key words: Emulsion; rheological properties; antioxidant activity; yield stress; cohesive energy; palm oil esters; tocotrienol

Palm oil is produced from the fruit of oil palm (*Elaeis guineensis*) which is grown in mass plantations in tropical countries such as Malaysia, Indonesia and Nigeria. The oil consists of 95% triglycerides and 5% diglycerides whereby carbons of the carboxyls range from 10–20 with or without double bonds (Tanaka *et al.* 2008). Palm oil esters (POEs) are a constituent of modified form of palm olein oil known simply as palm oil. Desirable characteristics of fat esters including non-toxicity, good fat solubility properties and excellent wetting at interfaces (Radzi *et al.* 2006) but without the greasy feeling when applied on the skin surface; these have attracted the attention of the industry. The emollient effect of POEs had been proven thereby making this oil highly recommendable for its incorporation into the topical preparation as oil phase. Thus, palm oil esters are excellent ingredient to be used in cosmeceutical and pharmaceutical formulations.

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Nanoemulsions are emulsion with droplet size in the range 20 nm-200 nm (Solans et al. 2003). They are independent of molecular size of the hydrophilic solute and the nature of the aqueous phase. In addition, nanoemulsions delivery system was independent of animal skin characteristics such as the stratum corneum thickness and the follicle-type (Wu et al. 2001). Thus, nanoemulsions due to their extremely small size are suitable to be used as delivery system in cosmeceuticals. However, nanoemulsions are only kinetically stable and therefore, it is also a very fragile system by nature (Tadros et al. 2004). As they are transparent and usually very fluid, the slightest sign of destabilization easily appears. They become opaque and creaming may be visible. Thus, stability of the nanoemulsion is a critical factor to be analysed. The achievement of developing long time stability of cosmetic products (3-years shelf life) is often difficult and deeply affects costs in the development of new formulations.

Rheology is an independent scientific discipline: studying the deformability, and flow properties of a matter under an applied stress or strain is revealed by McClements (1999). Owing to the fact that rheology can give a better picture of the behaviour of a material, it is therefore widely used as a tool to test the texture and flow behaviour of industrial products especially in the processing industries such as food (Lorenzo et al. 2008), cosmetics (Bummer & Godersky 1999), pharmaceuticals (Zumalacarregui et al. 2004), polymer (Karg et al. 1985), coating (Kikic et al. 1979), and oil processing (Martin et al. 2006). The rheological results also enable scientists to estimate the product's quality such as elasticity, viscosity, deformability, storage, shelf life including intermolecular interactions due to ultra-sensitivity at microstructure of materials.

Antioxidants neutralize damaging free radicals by quenching reactive molecules and, thus protecting cells from both endogeneous

2

stress (byproducts of cellular energy) and exogenous stressors (ultraviolet light, pollution, cigarette smoke etc.) (Choi & Berson 2006). Tocotrienol are fat-soluble vitamins related to the family of tocopherols. Tocopherol and tocotrienol are well recognized for their antioxidative effect (Kamal 1996). This effect depends primarily on the phenolic group in the chromanol ring, rather than the side chain (Burton & Ingold 1989). The trolox equivalent antioxidant capacity (TEAC) assay is widely applied to assess the amount of radicals that can be scavenged by an antioxidant, i.e. the antioxidant capacity (Lien *et al.* 1999).

The present investigation was focused on the preparation of palm esters-based emulsions of tocotrienol and to characterise the rheological properties of the emulsion systems. Furthermore, assessment of the *in vitro* antioxidant activity of esters-based palm was done by the TEAC assay.

EXPERIMENTAL

Materials

POEs was prepared in the laboratory according to the method of Keng *et al.* (2009) Sorbitan monooleate (Span[®] 80) and polyoxethylene (20) sorbitan monooleate (Tween[®]80) were purchased from Merck, Germany. The HLB values of sorbitan monooleate (Span[®] 80) and polyoxethylene (20) sorbitan monooleate (Tween[®] 80) are 4.3 and 15.0, respectively. Tocotrienol (Gold Tri. E 70) was from Golden Hope Bioganic, Malaysia. Xanthan gum from Xanthomonas campestris was obtained from Fluka Chemie GmbH, France. Freshly deionized water was obtained from water deionizer, Mili-Q (Milipore, USA).

Methods

Preparation of emulsions containing tocotrienol. Emulsions were formulated using POEs containing tocotrienol as dispersed oil phase and Mili-Q water as the continuous aqueous phase. Xanthan gum was dispersed in deionized water at 0.8% (w/w). Preparation of dispersed oil phase was performed by homogenizing 5% (w/w) of surfactants into oil phase with a Polytron homogenizer (Kinematica GmbH, Germany) rotor stator. The ratio of Span[®] 80:Tween[®] 80 was 1:4. The preparation was continued by adding the oil phase dropwise to the aqueous solution with continuous homogenized at 6000 r.p.m. for 5 min. The temperature was lowered to 40°C. At 40°C, the active ingredient was added. The emulsions were further homogenized using ultrasonic cavitation for 5 min. The sonifier tip horn was adjusted to 2 cm below the surface of a 100 ml sample.

Rheology measurement. A stress/rate controlled Kinexus Rheometer (Malvern Instrument, UK) with a temperature controller, was used to measure the rheological properties of the emulsion. The measurements were performed at $25.0 \pm 0.1^{\circ}$ C with $4^{\circ}/40$ mm cone and plate geometry. The samples were allowed to relax for 10 min after being loaded to the plate before the measurement was started.

In vitro antioxidant activity. The antioxidant activity was assessed as described below. Experiments were performed on the Varian Cary 50 UV-Vis spectrophotometer (Varian, Australia). Trolox (2.5, 5.0, 10.0, 15.0 µM) was prepared in ethanol. Ascorbic acid was prepared in 18 M Ω water to a concentration of 10.0, 15.0, 20.0 μ M and α -tocopherol in ethanol at 10.0, 15.0, 20.0 µM. ABTS, 2,2'-azinobis (3-ethylbenzothiazoline-6-sulphonic acid) diammonium salt was dissolved in water to a 7 mM concentration. ABTS radical cation (ABTS^{•+}) was produced by reacting ABTS stock solution with 2.45 mM potassium persulphate (final concentration) and allowing the mixture to stand in the dark at room temperature for 12 h-16 h before use. The radical was stable in this form for more than two days when stored in the dark at room temperature. ABTS.+ solution was then diluted with ethanol to an absorbance

of 0.70 (\pm 0.02) at 734 nm and equilibrated at 30°C (Roberta et al. 1999).

Diluted ABTS⁺ solution (1.0 ml) ($A_{734 nm} = 0.700 \pm 0.020$) was added to 10 µl of antioxidant compounds or Trolox standards in ethanol. The absorbance of the sample was taken at 30°C every min after initial mixing up to 6 min. An appropriate solvent blanks were run in each assay. All determinations were carried out three times, and in triplicate, on each occasion and at each separate concentration of the standard and samples. The percentage inhibition of absorbance at 734 nm was calculated and plotted as a function of concentration of antioxidants and of Trolox for the standard reference data (Roberta *et al.* 1999).

RESULTS AND DISCUSSION

Rheological Properties of Emulsions System

Steady-state flow: The sensitivity of emulsions to shearing. The sensitivity of these emulsions to shearing was tested in steadystate flow. The greater the yield stress σ_y the more brittle the emulsion, and this leads to believe that the emulsion either undergoes disorganization of its structure or takes longer to recover its initial states. Figures 1 and 2 summarize the yield stress data as a function of acoustic amplitudes (%) and surfactant concentration [% (w/w)], respectively. The vield stress of the emulsions increased with increasing acoustic amplitudes. The increase in acoustic amplitudes (20% to 100%) led to decrease in mean droplet size. The decrease of droplet size leads to the increase in the total droplet surface area. When the total surface area of the droplet increased, the strength of the attractive force will also increase. Thus, greater stress is required to initiate flow when high attractive force is holding the droplets resulting high viscosity with high yield stress (Pal 1996). Mean droplet size was another factor affecting the flow behaviour of the emulsion.

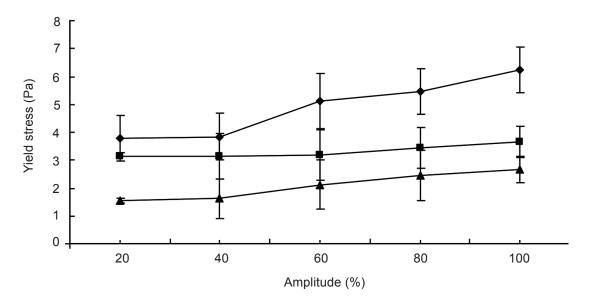


Figure 1. The yield stress of the emulsions as a function of acoustic amplitudes and oil phase concentration. Emulsions with 10% (----), 20% (----) and 30% (----) oil phase concentration.

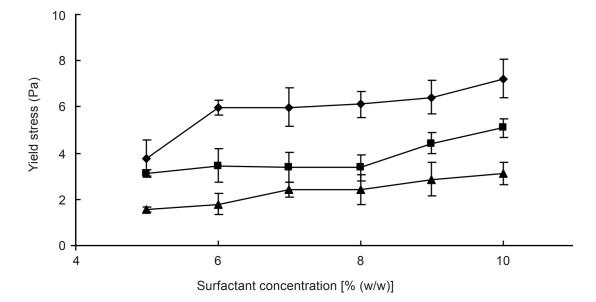


Figure 2. The yield stress of the emulsions as a function of surfactant concentration. Emulsions with 10%(-▲-), 20% (-▲-) and 30% (-▲-) oil phase concentration.

The yield stress of the emulsions increased with surfactant concentration (Figure 2) indicating structural integrity arising from the strong colloidal interaction between the droplets. The yield stress is the stress that has to be overcome before the emulsion starts to flow (Barnes 1999). The system with higher surfactant concentration tends to form a denser interfacial layer, which is incompressible (Hamill & Petersen 1966). Hence, the droplets in such sterically stabilized system are usually characterized as 'hard sphere' (McClements 1999). The strength of interaction forces (mainly the attractive and repulsive interactions) between the droplets for the hard sphere system (high surfactant concentration system) was relatively greater than the one with lower surfactant concentration. In the absence of the strong sterically repulsive effect, the droplets in the emulsions system with lower surfactant concentration were able to pack more efficiently even at low shear. Therefore, the droplets were aligning themselves easily with the shear field to initiate flow. This explained that the increase in surfactant concentration led to increase in yield stress of emulsion system.

Oil phase concentration in an emulsion system is another factor affecting the flow behaviour (Akhtar *et al.* 2005). The attractive force is one of the colloidal interactions which play an important role in the increase in viscosity and yield stress. The magnitude of viscosity and yield stress depend on the strength of the attractive force between the droplets (Pal 1996). Higher strength of attractive force between the droplets leads to increase in viscosity and yield stress.

Shear Stress versus Shear Rate Profile

The shear stress—shear rate profile of stabilized emulsions are depicted in *Figure 3*. Much like viscosity—shear rate profile, the rate of change of shear stress depends largely on the rate of change of shear rate. As depicted in *Figure 3*, at zero shear rate the shear stress responses are not zero. This suggested that these emulsions were shear thinning non-ideal plastic-like material, with yield stress (σ_y) response. In other words, they behaved like pseudoplastic material, which implied that flow can only be induced on these emulsions with the application of certain minimum amount of stress called yield stress. Figure 3 shows that above the yield stress these samples assume a linear shear stress—shear rate relationship. This in turn suggested that, these emulsions did not follow ideal Newtonian flow behaviour even at high shear rate domain. By contrast, the shear stress-shear rate relationship increased exponentially with a certain power law exponent at low-shear rate domain below yield stress, suggesting that the flow behaviour of these emulsions resembled that of plastic-like material at these low shearrate domain.

As far as the effect of concentration on yield stress was concerned, these profiles suggested that yield stress increased monotonically with surfactant concentration. This in turn indicated that all samples examined here exhibited non-Newtonian model type fluid behaviours, implying that the viscoelastic force dominated over the elastic force, and that the emulsions under investigation underwent structural deformation with shear rate irrespective of surfactant concentration. The increase in the yield stress as a function of surfactant concentration further indicated that emulsions with higher surfactant concentration possess higher degree of material structuring as opposed to lower surfactant concentration. This also means that emulsions with higher surfactant concentration offer a larger resistance to external force before they started flowing. This in turn suggested that emulsions stabilized with higher surfactant concentration undergo a greater degree of deformation under applied shear in comparison to emulsions stabilized with lower surfactant concentration.

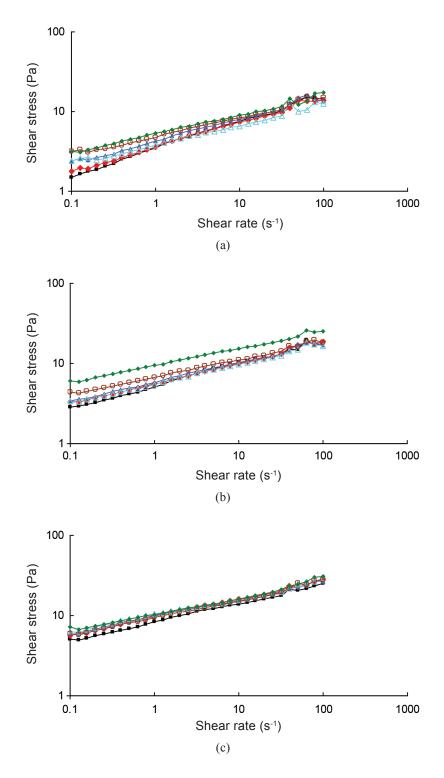


Figure 3. Effect of shear rate of emulsions on the shear stress for (a) 10% (b) 20% (c) 30% oil phase concentration. Surfactant concentration: 5% (→); 6% (→); 7% (→); 8% (→); 9% (→) and 10% (→).

Oscillatory Measurements: Strain Sweep Profile

A critical strain (γ_c) is the maximum applied strain where the emulsion still gives a linear response to shear stress with constant elastic modulus. The critical strain, γ_c of emulsions in different oil phase concentration is shown in *Figure 4*. The γ_c of the emulsions increased with the oil volume fraction and surfactant concentration. The γ_c was increased 50%, 46% and 86% as the surfactant concentration was increased from 5% to 10% (w/w) for emulsions with 30%, 20% and 10% (w/w) oil phase concentration, respectively. On the other hand, the γ_c increased more than 100% when the oil concentration was increased from 10% to 30% (w/w).

The increase of critical strain of emulsion with 30% (w/w) oil phase concentration when the surfactant concentration was increased implied that the highly packed droplets have developed a strong structure due to the high interdroplet interaction between the droplets which corresponded to the droplet size and droplet concentration of the emulsions system. Since the strength of the interdroplet interactions corresponded to the mean separation distance between the droplets, the highly packed emulsion system will therefore has greater interdroplet interaction forces. The high interdroplet interaction strength was able to hold the droplets and withstand the large deformation forces applied during the strain sweep test.

The strain sweep profiles also provided information about the elastic component of the emulsions. *Figures 5* and 6 show increasing trends in the elastic modulus (*G'*) of the emulsions with surfactant and oil concentration indicating that the interactions between droplets are relatively strong. A trend of increasing elastic modulus accompanying the increased of γ_c was observed. The cohesive energy (*E_c*) within the linear viscoelastic regime for when *G'* is in phase with the applied strain amplitude can also be obtained (Bossard *et al.* 2007), as shown below:

$$E_c = \int_0^{\gamma c} \sigma d\gamma \tag{1}$$

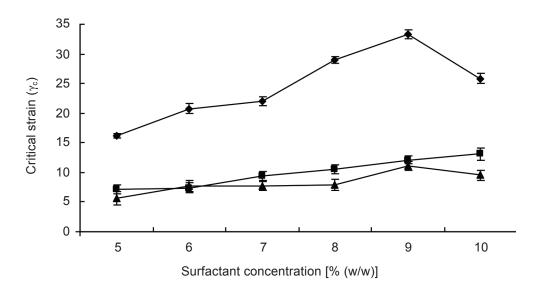


Figure 4. The critical strain, γ_c of the emulsions as a function of surfactant concentration. Emulsions with 10% (\rightarrow), 20% (\rightarrow) and 30% (\rightarrow) oil phase concentration.

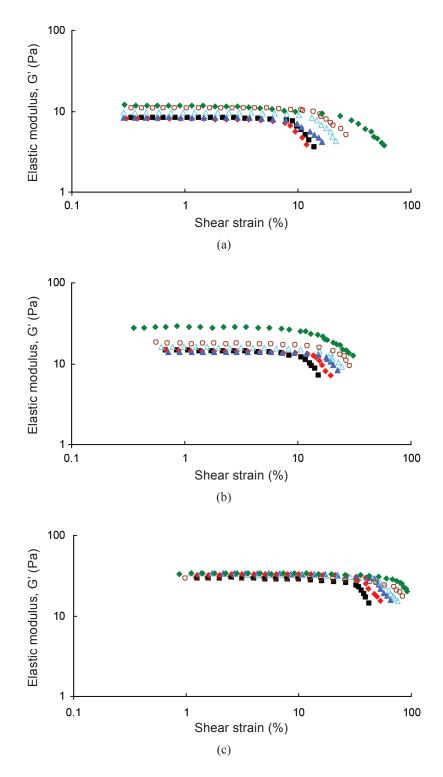


Figure 5. The linear viscoelastic region of the emulsions with a series of surfactant concentration $[5\% (\bullet)]; [6\% (\bullet)]; [7\% (\bullet)]; [8\% (\bullet)]; [9\% (\circ)] and [10\% (\bullet)] for (a) 10\%, (b) 20\% and (c) 30\% oil phase concentration.$

When σ equals G' in the linear viscoelastic region, the cohesive energy is defined as:

$$E_c = \frac{1}{2} \gamma^2_{\ c} G' \tag{2}$$

Tadros (2004) explained that the cohesive energy was related to the structure of the emulsion system, which correlated to the droplet size and number of contact area between the droplets. The droplet concentration and the packing of the droplets influenced the strength of the cohesive force. As discussed before, the number of droplets was increased as the oil concentration was increased from 10% to 30% (w/w) at fix surfactant concentration. As a result, the number of contacts area within the droplets increased. Thus, increases in the cohesive energy of the emulsions system were observed.

 E_c ranged from a low 0.13 J/m³ for emulsion with 5% (w/w) surfactant concentration in 10% (w/w) oil phase concentration to a high of 15.88 J/m³ for emulsion with 10% (w/w) surfactant concentration in 30% (w/w) oil phase concentration. E_c was low for emulsion with 5% (w/w) surfactant concentration in 10% (w/w) oil phase concentration as the elasticity was low (*Figure 6*). The higher the cohesive energy, the more stable a system was as the elastic strength was basically a measure of the strength of the internal structure. This in turn demonstrated that the emulsion samples under examination were stable systems, and that the stability of these emulsions systems was enhancing with decreasing droplet size.

Figure 7 show that the cohesive energy increased significantly with surfactant concentration especially for the emulsions with 30% (w/w) oil phase concentration. The dramatic increase of cohesive force was due to the highly packed systems related to the interdroplet interactions that had been previously discussed.

In vitro Antioxidant Activity

The concentration-response curve for six sequentially and separately prepared stock standards of Trolox was illustrated in

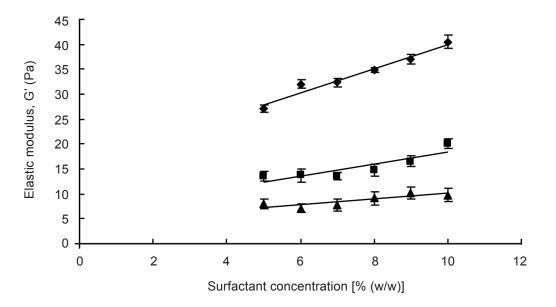


Figure 6. The elastic modulus of the emulsions as a function of surfactant concentration. Emulsions with 10% (--), 20% (--) and 30% (--) oil phase concentration.

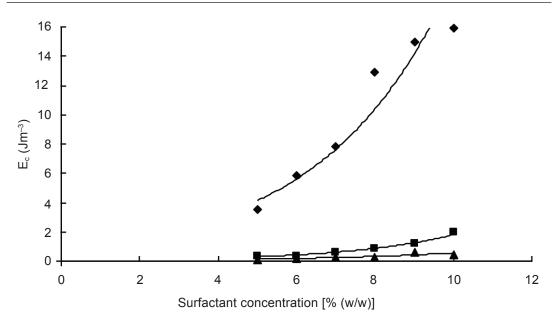


Figure 7. The cohesive energy of the emulsions as a function of surfactant concentration and oil concentration. Emulsions with 10% (\rightarrow), 20% (\rightarrow) and 30% (\rightarrow) oil phase concentration.

Figure 8. Trolox, the water-soluble analogue of a-tocopherol was known for its high radical scavenging activity and was therefore frequently used as a model compound. It was commonly used as a reference antioxidant, in which the radical scavenging activity was expressed as Trolox equivalents. The higher the concentration of Trolox used, the more the absorbance of ABTS radicals was suppressed. The selected absorption wavelength was at 734 nm because ABTS*+ has maximum absorption at 734 nm and most antioxidant does not absorb light at 734 nm. Figure 9 illustrates the effects of the duration of interaction of specific antioxidants on the suppression of the absorbance of the ABTS^{•+} radical cation at 734 nm for Trolox, the standard reference compound, compared with α -tocopherol, ascorbic acid and emulsion samples containing tocotrienol.

The results demonstrated that the reaction with ABTS^{•+} was completed after 1 min. 30% dispersed phase emulsion containing tocotrienol (15 μ M) has minimum absorption than in 20%

tocotrienol (15 µM) with ABTS^{•+} within 6 min. The absorption at wavelength 734 nm for ABTS⁺⁺ radical cation was 0.73 followed by ABTS⁺⁺ radical cation in ascorbic acid and tocopherol which gave 0.42 and 0.28, respectively. From the results, 30% dispersed phase emulsion containing to cotrienol $(15 \,\mu\text{M})$ revealed better antioxidant properties by presenting lower absorbance value as compared the standard reference compound, Trolox. The extent of inhibition of the absorbance of the ABTS^{•+} was plotted as a function of concentration in order to determine the TEAC that can be assessed as a function of time. The dose-response curve obtained by analysis of a range of concentrations of antioxidant compounds and Trolox standards was plotted as the percentage inhibition of the absorbance of the ABTS*+ solution as a function of concentration of antioxidant (Figure 10). The result showed that 30% dispersed phase emulsion containing tocotrienol (15 µM) has an inhibitory effect, a maximum concentration of 20 μM reducing ABTS^{•+} by about 93%.

and 10% dispersed phase emulsion containing

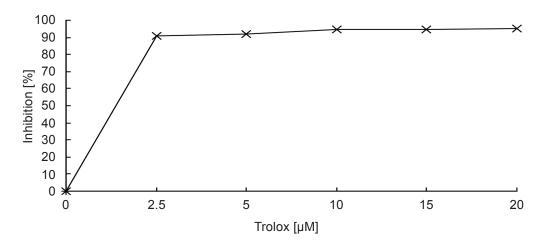


Figure 8. Concentration-response curve for the absorbance at 734 nm for ABTS⁺⁺ *as a function of concentration of standard Trolox solution.*

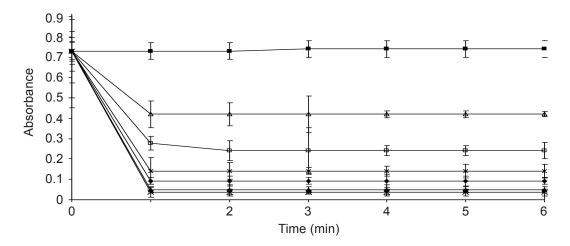


Figure 9. Effects of time on the suppression of the absorbance of the ABTS⁺⁺; ABTS⁺⁺ (---);
ABTS⁺⁺ + ascorbic acid (---); ABTS⁺⁺ + tocopherol (---); ABTS⁺⁺ + Trolox (---); ABTS⁺⁺ + 10% disperse phase containing tocotrienol (---); ABTS⁺⁺ + 20% disperse phase containing tocotrienol (---).
The concentration of the samples used in the determination is 15 μM.

From the result, 20% dispersed phase emulsion containing to cotrienol (15 μ M) appears to exert a slightly greater inhibitory effect on the ABTS⁺⁺ than on that of 10% dispersed phase emulsion. The presence of the 30% dispersed phase emulsion containing to cotrienol (15 μ M) enhanced its inhibitory effect on the ABTS⁺⁺ since 20 μ M of 20% dispersed phase emulsion containing to cotrienol (15 μ M) and 10% disperse phase emulsion containing to cotrienol (15 μ M) only produced 87.5% and 82.8% inhibition; respectively of the ABTS as say system. The antioxidant activity could be expressed in terms of the total contribution to the antioxidant activity over the time range studied by calculating the TEAC, the gradient of the plot of the percentage inhibition of absorbance vs. concentration plot for the antioxidant in question is divided by the gradient of the plot for Trolox. This gives the TEAC at the specific time points, 1 min, 4 min and 6 min (*Figure 11*).

From the results, it was found that all samples tested were able to scavenge ABTS⁺⁺ radical cation. Their antioxidant activities

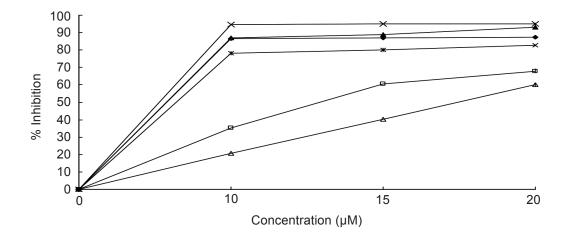


Figure 10. Effects of concentration of the antioxidant on the inhibition of the ABTS⁺⁺; ABTS⁺⁺ + ascorbic acid (→); ABTS⁺⁺ + tocopherol (→); ABTS⁺⁺ + Trolox (→); ABTS⁺⁺ + 10% dispersed phase containing tocotrienol (→); ABTS⁺⁺ + 20% dispersed phase containing tocotrienol (→) and ABTS⁺⁺ + 30% dispersed phase containing tocotrienol (→). The concentration of the samples used in the determination is 15 µM.

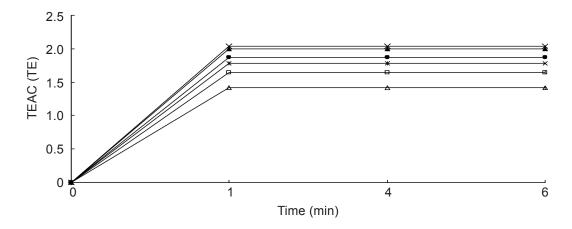


Figure 11. Profile of the variation of gradient of the percent inhibition vs. concentration plot of each antioxidant at 1, 4 and 6 min: $ABTS^{+} + ascorbic acid (- -); ABTS^{+} + tocopherol (--); ABTS^{+} + Trolox (--); ABTS^{+} + 10\% dispersed phase containing tocotrienol (--); ABTS^{+} + 20\% dispersed phase containing tocotrienol (--); The concentration of the samples used in the determination is 15 <math>\mu$ M.

measured at 6 min and expressed as the TEAC value or IC₅₀ were relatively low as compared to Trolox. The higher TEAC values implied higher antioxidant capability. The TEAC at 6 min of 30% dispersed phase emulsion containing tocotrienol (15 μ M) was 2.00 whereas the TEAC values of tocopherol and ascorbic acid were 1.64 and 1.42 respectively. Tocotrienol in 30% dispersed phase appeared to have a much higher TEAC value than tocopherol and ascorbic acid in 15 μ M concentrations, respectively. This means that tocotrienol can be qualified as the best antioxidant compared to tocopherol and ascorbic acid with the TEAC assay.

CONCLUSIONS

The results obtained in the present work showed that emulsions containing palm esters-based as oil phase was a suitable carrier system for the incorporation of tocotrienol with reasonably high values of yield stress, suggesting the stabilization of emulsions. *In vitro* antioxidant activity using TEAC assay was applied to assess the total amount of radicals that could be scavenged by antioxidant samples. The findings indicated that tocotrienol in emulsion with 30% (w/w) dispersed phase exhibited greater inhibitory effects on the ABTS⁺. Results were encouraging and it substantiated the role of palm esters-based emulsions containing tocotrienol as an effective antioxidant therapy.

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Pd(II) Complexes with Nitrogen-oxygen Donor Ligands: Synthesis, Characterization and Catalytic Activity for Suzuki-Miyaura Cross-coupling Reaction

M. T. AMALINA¹, B. HADARIAH^{1*}, K. KARIMAH¹ AND W. I. W. NAZIHAH¹

Inexpensive bidentate Schiff base ligands (O1, O3) and their Pd(II) complexes (OPd1, OPd3) were successfully synthesized and characterized using CHN elemental analysis, FTIR, ¹H and ¹³C NMR, melting point determination, molar conductivity and magnetic moment. It revealed that these Schiff bases behaved as bidentate ligands in their complexes. The spectral data indicated that the ligands co-ordinated through the phenolic oxygen and the azomethine nitrogen atoms. Magnetic moment data suggested the existence of square planar Pd(II) complexes, while non-electrolytic behaviour indicated the absence of counter ions in chloroform. The Pd(II) complexes showed good catalytic activities for Suzuki-Miyaura cross-coupling reaction between iodobenzene with phenylboronic acid at 1.0 mmol% catalyst loading.

Key words: Bidentate Schiff base; nitrogen-oxygen donor ligands; Pd(II); Suzuki-Miyaura crosscoupling reaction; synthesis; physicochemical properties; catalytic activity; characterization

Schiff base ligands are considered 'privileged ligands' and are attractive because they are easily prepared by the condensation between aldehyde/ketone and imines both of which are relatively cheap and easily available. Stereogenic centres or other elements of chirality such as planes and axes can be introduced in the synthetic design (Cozzi 2004) as well as benzene rings containing electron donating or withdrawing substituents. The mono-, di-, tri- and multi-dentate chelating Schiff base ligands can be designed according to the binding environments of metal ions. The metal complexes of chiral Schiff base ligands showed stereoselectivity in organic transformation, hence the synthesis of chiral complexes become an important area of current research in co-ordination chemistry (Gupta & Sutar 2008).

Schiff base ligands are able to co-ordinate many different metals (Osman 2006; Sallam 2006) and to stabilize them in various oxidation states. The Schiff base complexes have been used in catalytic reactions (Dhara *et al.* 2010; Tamizh & Karvembu 2012) and as models for biological systems (Singh *et al.* 2012; Mohamed *et al.* 2010). Many Schiff base complexes show excellent catalytic activity in various reactions at high temperature (>100°C) and in the presence of moisture (Gupta & Sutar 2008).

The Suzuki-Miyaura cross-coupling reaction is a powerful method for the synthesis of biaryl bonds. The importance of biaryl units as molecular components in pharmaceuticals, herbicides and natural products, as well as in engineering materials such as conducting polymers, molecular wires and liquid crystals,

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has attracted enormous interest (Paul & Clark 2003). Numerous variants (Gillis & Burke 2009), optimizations and applications (McGlaken & Fairlamb 2009) have been disclosed in the literature. The robust nature of this reaction has led to its widespread use in the pharmaceutical industry (Torborg & Beller 2009). The relative thermal stability, insensitivity to air or moisture, and low toxicity (Miyaura & Suzuki 1995) of boronic acid constitute a highly valuable practical advantage for both academic and industrial applications.

As a part of our interest in designing new, inexpensive ligands and studying their co-ordination behaviour and catalytic application, we report herein the synthesis and characterization of a new type of ligands and their Pd(II) complexes as catalysts in Suzuki-Miyaura cross-coupling reaction.

MATERIALS AND METHODS

Experimental Sections

Materials. All reagents and chemicals used in this investigation were laboratory pure grade available from Acros, Merck and Sigma-Aldrich. The solvents for the spectral study were spectroscopic grade and used without further purification.

Techniques. Microanalyses for carbon, hydrogen and nitrogen were determined using a Thermo Finnigan Flash Elemental Analyzer 2000. The IR spectra were obtained on a Perkin Elmer 1750X FTIR spectrophotometer $(4000 \text{ cm}^{-1} - 400 \text{ cm}^{-1})$ with samples prepared as KBr pellets. Melting points of the products were determined using Buchii-B454 and are uncorrected. Proton (1H) and carbon (13C) NMR (300 MHz) spectra were recorded on a Bruker Varian spectrometer in CDCl₃ and reported in p.p.m. (δ) from the internal standard TMS. The magnetic susceptibilities were determined on Sherwood Auto Magnetic Susceptibility Balance at room temperature (25°C) using $Hg[Co(SCN)_4]$ as a calibrant; the diamagnetic corrections were calculated from Guoy method. Molar conductance measurements of freshly prepared Schiff base ligands and their transition metal complexes solutions were determined in chloroform ($\sim 10^{-3}$ M) at room temperature using a Mettler Toledo Inlab 730 conductivity meter. The formation of products from catalytic testing was monitored using Gas Chromatography (GC) technique (Bhunia *et al.* 2010). Yields were calculated for a specific set of parameters at a specific time according to the product ratios.

Synthesis of Schiff base ligands.

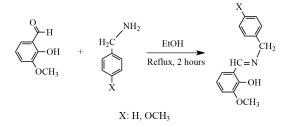


Figure 1. General reaction for the synthesis of Ovan-series ligands.

Synthesis of (E)-2-[(benzylimino)methyl]-6methoxyphenol [O1]

To *o*-vanillin (50 mmol, 7.6091 g) was dissolved in ethanol (25 ml), an ethanolic solution of benzylamine (50 mmol, 5.3869 g) was added and the mixture was stirred for overnight. The ethanol was allowed to evaporate slowly at room temperature. Orange-coloured solid product was evident after a week. The solid residue was filtered, washed with ice-cold ethanol and air-dried at room temperature. The yield was 36.8%.

Synthesis of (E)-2-methoxy-6-[(4-methoxybenzylimino)methyl]phenol [O3]

To *o*-vanillin (2 mmol, 0.3075 g) was dissolved in ethanol (5 ml), an ethanolic solution of 4-methoxybenzylamine (2 mmol, 0.2744 g) was added dropwise and the mixture was heated under reflux for 2 h. Upon cooling, there was no formation of solid materials. Ethanol was removed using rotary evaporator. The ligand was used in complexation with Pd(II) metal salt.

Synthesis of Pd(II) complexes.

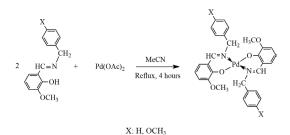


Figure 2. General reaction for the synthesis of Pd-Ovan-series complexes.

<u>Synthesis of Bis(2-((E)-(benzylimino)methyl)-</u> <u>6-methoxyphenoxy)palladium(II) [OPd1]</u>

The ligand, O1, (5 mmol, 1.2064 g) was dissolved in acetonitrile (10 ml) in a roundbottomed flask. Palladium(II) acetate (2.5 mmol, 0.5612 g) was dissolved separately in acetonitrile (10 ml) and added into the flask containing the ligand solution. The mixture was stirred and refluxed for 4 h upon which brown solids were formed. It was isolated by gravity filtration, washed with ice-cold acetonitrile and air dried at room temperature. The yield was 90.6%.

<u>Synthesis of Bis(2-methoxy-6-((E)-(4-methoxybenzylimino)methyl)phenolato)</u> palladium(II) [OPd3]

The ligand, O3, (2 mmol, 0.5426 g) was dissolved in acetonitrile (5 ml) in a roundbottomed flask. Palladium(II) acetate (1 mmol, 0.2246 g) was dissolved separately in acetonitrile (5 ml) and added into the flask containing the ligand solution. The mixture was stirred and refluxed for 4 h upon which a brown solid was formed. It was isolated by gravity filtration, washed with ice-cold acetonitrile and air dried at room temperature. The yield secured was 80.8%. General reaction of Suzuki-Miyaura crosscoupling reaction. The palladium(II) Schiff base complexes were tested as homogeneous catalysts in a series of Suzuki-Miyaura crosscoupling reaction, between iodobenzene and phenylboronic acid to produce biphenyl. The general procedure (*Figure 1*) is as follows: Iodobenzene (1 mmol), phenylboronic acid (2 mmol), triethylamine, Et₃N (2.4 mmol), palladium(II) Schiff base (0.01 mmol) and solvent DMA (7 ml) were mixed in Radley's 12-placed reaction carousel and refluxed whilst being purged with nitrogen. The reaction was monitored every 6 h and sampling was done at 6 h, 12 h and 24 h.

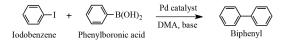


Figure 3. Catalytic reaction of iodobenzene with phenylboronic acid.

RESULTS AND DISCUSSION

Physicochemical Properties of the Synthesized Compounds

All of these bidentate Schiff base ligands and their Pd(II) complexes were intensely coloured, air and moisture free. The ligands and their metal complexes were very stable solids at room temperature, without undergoing decomposition. Yields of the complexes were higher than those of the ligands. The ligands had very high solubility in almost all polar solvents attributable to their polar nature. The complexes however, had very low solubility in polar organic solvents such as Et₃OH, MeOH, DMSO, CH₂Cl₂, etc. but soluble in relatively less polar CHCl₃. These synthesized ligands contained both polar and non-polar groups such as -OH, -OCH₃, -CH₃ etc. The C, H and N percentages were theoretically calculated and the measured values were in accordance to the suggested formula. The physical and analytical data of the Schiff base ligands and their complexes are shown in Table 1.

| Ligand/ | Empirical | Empirical Colour Melting point Elemental Analysis (%) (Foun | | | (Found) | |
|---------|---|---|---------|----------------------|--------------------|--------------------|
| Complex | formula | Coloui | (°C) | С | Н | Ν |
| 01 | $C_{15}H_{15}NO_2$ | Yellow | 62–65 | 74.67 (74.77) | 6.27 (6.31) | 5.81 (5.91) |
| OPd1 | $\mathrm{C}_{30}\mathrm{H}_{28}\mathrm{N}_{2}\mathrm{O}_{4}\mathrm{Pd}$ | Brown | 259–261 | 61.39 (61.47) | 4.81 (4.79) | 4.77 (4.74) |
| O3 | $C_{16}H_{17}NO_3$ | Yellow | _ | 70.83 (71.08) | 6.32 (6.32) | 5.23 (5.23) |
| OPd3 | $C_{32}H_{32}N_2O_6Pd$ | Brown | 204–205 | 59.40 (59.12) | 4.98 (4.99) | 4.33 (4.35) |

Table 1. Physical and analytical data for ovan-series ligands and their complexes.

IR Spectral Studies

In general, all infrared spectra presented the same general characteristics. The appearance of the C=N (azomethine) peaks in the range 1620–1634 cm⁻¹ could be clearly seen in the spectra, indicating the formation of the Schiff bases. The broad band at 2300 cm⁻¹-3300 cm⁻¹ could be attributed to the intramolecular hydrogen-bonded O-H group (Zolezzi et al. 1999). This band was absent in the spectra of complexes due to deprotonation of the phenolic moiety upon complexation. The Pd(II) ion was co-ordinated through the nitrogen and oxygen atoms of the hydroxyl group. The azomethine C=N bands were seen to be shifted to lower frequencies, 1612 cm⁻¹–1623 cm⁻¹ in all Pd(II) complexes due to the withdrawal

of electron density from the nitrogen atom owing to co-ordination (Zolezzi et al. 1999). A similar effect was observed in the stretching vibration of the Schiff base phenolic C-O and methoxy groups, with respect to the same group in the complexes where it was shifted to a lower frequency, strongly indicating oxygen co-ordination to the metal centre. The appearance of new bands at 462 cm⁻¹–544 cm⁻¹ and 581 cm⁻¹-660 cm⁻¹, that ascribed Pd-O and Pd-N vibrations, support the evidence of the participation of the nitrogen atom of the azomethine group and oxygen atom of the of OH group of the ligand in the complexation with metal ions (Ouf et al. 2010; Mustafa et al. 2009). The significant bands of ligands and their Pd(II) complexes and their spectra are summarized in *Table 2* and *Figure 4*.

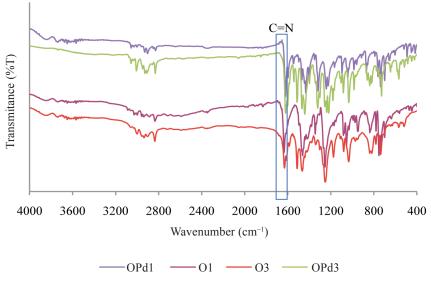


Figure 4. FTIR Spectra of O1, O3, OPd1 and OPd3.

| | | | 0 | | | |
|---------|-------------------------------|-----------------|-----------------|---------------------|----------------------|---------------------|
| Ligand/ | Frequency (cm ⁻¹) | | | | | |
| Complex | v(C=N) | v (C–N) | v (C–O) | $\mathbf{v}(OCH_3)$ | v (C-OH) | v (M–O, M–N) |
| 01 | 1634 <i>(s)</i> | 1344 <i>(w)</i> | 1254 <i>(s)</i> | 1054 <i>(w)</i> | 2300-3300 <i>(b)</i> | - |
| OPd1 | 1622 <i>(s)</i> | 1316 <i>(s)</i> | 1241 <i>(s)</i> | 1095 <i>(w)</i> | _ | 657 416 <i>(w)</i> |
| O3 | 1630 <i>(w)</i> | 1344 <i>(w)</i> | 1251 <i>(s)</i> | 1033 <i>(w)</i> | 2500-3200 <i>(b)</i> | _ |
| OPd3 | 1620 <i>(s)</i> | 1320 <i>(s)</i> | 1239 <i>(s)</i> | 1032 <i>(w)</i> | _ | 567 515 <i>(w)</i> |

Table 2. Infrared data for ligands and their Pd(II) complexes.

(s) = Strong; (b) = Broad; (w) = Weak.

¹H and ¹³C NMR Studies

In the ¹H NMR of the ligands (*Table 3*), the chemical shift for methylene appear at 4.74 p.p.m.–4.82 p.p.m. as singlets and slightly shifted to higher values of 5.04 p.p.m.-5.12 p.p.m. in their respective Pd(II) complexes. The methoxy peaks for ligands appear at 3.88 p.p.m.-3.90 p.p.m. while in complexes, they appear at 3.75 p.p.m.-3.76 p.p.m., slightly shifted to lower values. Upon co-ordination, there is an upfield shift of peaks for azomethine protons from 8.38 p.p.m.-8.42 p.p.m. to 7.69 p.p.m.-7.72 p.p.m.. This upfield shift is attributed to the conformational change that occurs in the ligand upon chelation (Mahamo et al. 2012). The phenolic proton appears at 13.85 p.p.m. in O1 and

disappeared upon complexation, indicating that the complexation was successfully achieved through deprotonation of the phenol. The OH peak in O3 that was expected to appear in the downfield region (10 p.p.m.–14 p.p.m.) (Saheb & Sheikhshoaie 2011) of the spectra was not observed as the labile phenolic proton may have undergone a rapid exchange with the deuterium in the solvent.

The ¹³C singlet signals for the imine carbons (C=N) in complexes are found in the region 162.3 p.p.m.–162.8 p.p.m. (Table 4). This is an upfield shift from 165.2 p.p.m.–165.6 p.p.m. observed for the free ligands, further affirming the co-ordination of the ligand through the imine group to the metal centre. There is

| Assistants | Chemical shifts (p.p.m.) | | | | | |
|--|--------------------------|----------------------|----------------------|----------------------|--|--|
| Assignments | O1 | OPd1 | O3 | OPd3 | | |
| $C-\underline{C}H_2$ | 4.82 <i>(s)</i> | 5.12 <i>(s)</i> | 4.74 <i>(s)</i> | 5.04 <i>(s)</i> | | |
| N= <u>C</u> H | 8.42 <i>(s)</i> | 7.72(s) | 8.38 <i>(s)</i> | 7.69(s) | | |
| <u>C</u> ³ -H (Ar) | 6.84–6.89 <i>(d)</i> | 6.76–6.81 <i>(d)</i> | 6.92–6.93 <i>(d)</i> | 6.78–6.79 <i>(d)</i> | | |
| <u>C</u> ⁴ -H (Ar) | 6.81–6.84 <i>(t)</i> | 6.47–6.52 <i>(t)</i> | 6.86–6.89 <i>(t)</i> | 6.83–6.84 <i>(t)</i> | | |
| <u>C</u> ⁵ -H (Ar) | 6.89–6.91 <i>(d)</i> | 7.26–7.29 <i>(d)</i> | 7.21–7.24 <i>(d)</i> | 7.36–7.39 <i>(d)</i> | | |
| <u>C</u> ¹³ -H (Ar) | 6.91–7.26 <i>(t)</i> | 7.29(t) | _ | _ | | |
| $\underline{\mathbf{C}}^{11}, \underline{\mathbf{C}}^{15}$ -H (Ar) | 7.32(<i>d</i>) | 7.44–7.46 <i>(d)</i> | 6.76–6.79 <i>(d)</i> | 6.47–6.52 <i>(d)</i> | | |
| $\underline{\mathbf{C}}^{12}, \underline{\mathbf{C}}^{14}$ -H (Ar) | 7.26–7.32 <i>(t)</i> | 7.22–7.26 <i>(t)</i> | 7.29–7.30 <i>(d)</i> | 6.77–6.86 <i>(d)</i> | | |
| $C^{13}-OCH_{3}$ | _ | _ | 3.79 <i>(s)</i> | 3.77(s) | | |
| $Ar-O\underline{C}H_3$ | 3.90 <i>(s)</i> | 3.75 <i>(s)</i> | 3.88 <i>(s)</i> | 3.76(s) | | |
| <u>С</u> ¹-ОН | 13.85 <i>(b)</i> | _ | N.D. | _ | | |

Table 3. Chemical shift of ¹H NMR for ligands and their Pd(II) complexes.

(s) = Singlet; (d) = Doublet; (t) = Triplet; (b) = Broad; N.D. = Not detected; Ar = Aromatic.

| A | Chemical shifts (p.p.m.) | | | |
|---|--------------------------|------------------|------------------|------------------|
| Assignments | 01 | OPd1 | 03 | OPd3 |
| С- <u>С</u> Н ₂ | 62.68 <i>(s)</i> | 58.85 <i>(s)</i> | 64.40 <i>(s)</i> | 58.33 <i>(s)</i> |
| N= <u>C</u> H | 165.6 <i>(s)</i> | 162.8 <i>(s)</i> | 165.2 <i>(s)</i> | 162.3 <i>(s)</i> |
| <u>C</u> ³ -H (Ar) | 113.9 <i>(s)</i> | 114.0 <i>(s)</i> | 114.0 <i>(s)</i> | 113.8 <i>(s)</i> |
| <u>C</u> ⁴ -H (Ar) | 117.9 <i>(s)</i> | 120.2(s) | 117.8 <i>(s)</i> | 113.8 <i>(s)</i> |
| <u>C</u> ⁵ -H (Ar) | 122.9 <i>(s)</i> | 125.4 <i>(s)</i> | 122.9 <i>(s)</i> | 125.4 <i>(s)</i> |
| <u>C</u> ¹³ -H (Ar) | 127.3 <i>(s)</i> | 127.1 <i>(s)</i> | 158.8 <i>(s)</i> | 158.8 <i>(s)</i> |
| <u>C</u> ¹¹ , <u>C</u> ¹⁵ -H (Ar) | 127.5 <i>(s)</i> | 127.1 <i>(s)</i> | 129.9 <i>(s)</i> | 129.8 <i>(s)</i> |
| <u>C</u> ¹² , <u>C</u> ¹⁴ -H (Ar) | 128.6 <i>(s)</i> | 128.4 <i>(s)</i> | 114.2 <i>(s)</i> | 114.0 <i>(s)</i> |
| $C^{13}-OCH_{3}$ | _ | _ | 55.30 <i>(s)</i> | 55.24 <i>(s)</i> |
| $Ar-O\underline{C}H_3$ | 56.01 <i>(s)</i> | 55.90 <i>(s)</i> | 56.04 <i>(s)</i> | 55.95 <i>(s)</i> |
| <u>C</u> ¹ -OH | 151.7 <i>(s)</i> | - | 151.9 <i>(s)</i> | - |

Table 4. Chemical shift of ¹³C NMR for ligands and their Pd(II) complexes.

(s) = Singlet; Ar = Aromatic.

also an upfield shift from 62.68 p.p.m.-64.40 p.p.m. to 58.33 p.p.m.-58.85 p.p.m. and from 56.01 p.p.m.-56.04 p.p.m. to 55.90 p.p.m.-55.95 p.p.m. with respect to the free ligand in the signals for the methylene and methoxy carbons, respectively, upon co-ordination to the metal centre as per reported by Mahamo *et al.* (2012) and Dilworth *et al.* (1994). In the free ligands, C-OH signals appear at 151.7 p.p.m.-151.9 p.p.m. (Şenol *et al.* 2011) and disappeared upon complexation where the phenolic moiety was deprotonated.

Molar Conductance and Magnetic Moment

The molar conductance values of the Pd(II) complexes was found to be 0 Ω^{-1} cm² mol⁻¹ suggesting their non-electrolytic nature (Ben-Saber *et al.* 2005). The magnetic moment of the complexes revealed their diamagnetic nature where $\mu_{eff} = 0$, consistent with the expected square planar geometry (Raman *et al.* 2007).

Catalytic Activity

The Pd(II) complexes namely OPd1 and OPd3 were tested as catalysts in the Suzuki-Miyaura cross-coupling reaction of iodobenzene with phenylboronic acid in the presence of triethylamine (Et₃N) as base in N₃N-dimethylacetamide (DMA) at 100°C. Et₃N that is soluble in the reaction mixture was a good base for the reaction due to its capability to give high conversion, although in its presence trace quantities of palladium metal could occasionally be observed as a precipitate against the walls of the glass tubes.

In most of the cases, Et₃N was the base of choice to activate the boron species in order to increase its nucleophilicity and give a clean reaction. This is because the organoborons compounds are highly covalent in character (Matos & Soderquist 1998). Solubility of the bases plays a great rule in Suzuki-Miyaura coupling reaction (Papp *et al.* 2006).

The reaction was monitored using GC-FID by percentage conversion of iodobenzene at every 3 h and sampling was done at 3, 6, 9, 12 and 24 h. A control reaction without catalyst had been set up and there was no indication of iodobenzene conversion after 24 h.

The results are summarized in the *Figure 5*. It is observed, with increasing time, the %

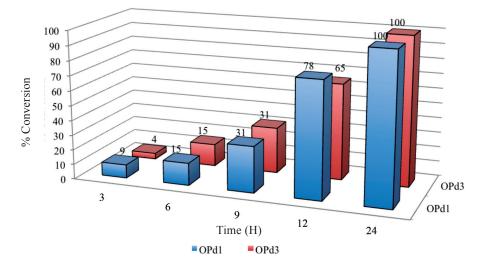


Figure 5. Percentage conversion of Iodobenzene using OPd1 and OPd3 as catalysts within 24 h.

conversion of iodobenzene also increased. OPd1 showed its ability to convert iodobenzene faster than OPd3 in the early hours. At the end of the reaction, phenylboronic acid was found to couple smoothly with iodobenzene providing excellent yields up to 100% after 24 h for both catalysts.

CONCLUSION

Two ligands and their Pd(II) complexes were successfully synthesized as confirmed by the characterization via various physicospectral techniques. The Schiff base ligands co-ordinated through phenolic oxygen and azomethine nitrogen atoms as bidentate chelates as indicated by the spectral data. It was observed that both Pd(II) complexes, OPd1 and OPd3 displayed properties of good catalysts for the reaction, with up to 100% conversion of iodobenzene after 24 h of reaction time at 100°C in inert conditions.

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Cigarette Smoking among Male Teenagers in Malaysia — A Narrative Review

R. III P. DIOSO

.This narrative review of a contemporary healthcare issue focuses on strategies and healthcare initiatives to curtail the increasing prevalence of active cigarette smoking among male teenagers in Malaysia — a mission of the Malaysian Millennium Development Goal and the United Nations Development Programmes on all health sectors. This narrative review analysed global and Malaysian views on cigarette smoking.

Key words: Healthcare; strategies; initiatives; curtail cigarette smoking; male teenagers; Malaysia

This narrative review aims to analyse the impact of active cigarette-smoking as a contemporary healthcare issue in Malaysia. Another aim of this review is to identify the best strategy and healthcare initiative to curtail the prevalence of cigarette-smoking in all health sectors.

According to World Health Organisation (WHO 2012), cigarette smoking is the action or habit of inhaling and exhaling the smoke of cigarettes. Smoking in general can attribute to a lot of physical illnesses and diseases; hence, it distracts the mental, psychological and social capacity of an individual to adapt (Kiechl *et al.* 2002; Lim *et al.* 2010; Isohanni *et al.* 2006; Lee *et al.* 2005; Naing *et al.* 2004; WHO 2012; Ferrante *et al.* 2010). Specifically, active cigarette-smoking disturbs a person's well being (Mohide, 1988; Kiechl *et al.* 2002) because it weakens their heart, brains and lungs.

There are two types of cigarette smoking. The active smoking or first-hand smoking is known as the first type defined as, the act of inhaling the smoke of cigarettes from one's self (Kiechl *et al.* 2002; Lim *et al.* 2010; Isohanni *et al.* 2006; Lee *et al.* 2005; Naing *et al.* 2004; WHO, 2012; Ferrante *et al.* 2010). The second type is called the second-hand smoking or passive smoking defined as, the exposure to environmental cigarette smoke (Kiechl *et al.* 2002; Lim *et al.* 2010).

Search Strategy

Literatures reviewed were from years 2000–2014. Limitations were set on databases to English language. Key words used were: *Malaysian male teenage cigarette smokers; active cigarette smoking habits; prevalence of male cigarette smoking in Malaysia and worldwide;* and *severity of the prevalence of active cigarette smoking among male teenagers,* that were entered as text in search engines such as *Researchgate, Google Scholars* and *Cochrane databases* with abstracts and full texts.

Methods of Review

A critiquing framework (*Figure 1*) was used to analyse the literatures selected for this review since it was designed with nine categories

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| Quantitative | Qualitative |
|------------------------------|---|
| Research design | Philosophical background |
| Experimental hypothesis | Research design |
| Operational definitions | Concepts |
| Population - | Context |
| Sample | Sample |
| Sampling | Sampling |
| Validity/reliability of data | Auditability of data |
| Data analysis 🔺 🔸 | Credibility/comfirmability of data analysis |
| Generalizability | Transferability |

Common features of:

Figure 1. Critiquing framework (Couglan et al. 2007; Ryan et al. 2007).

as guides to conveniently aid the reviewer to make sense of evidence from clinical trials and theories (Polit & Beck 2011; Ryan *et al.* 2007; Couglan *et al.* 2007).

RESULT

Worldwide Prevalence of Cigarette Smoking

A World Health Organisation (WHO, 2012) study has showed a total of 43 trillion active cigarette smokers all over the world (*Figure 2*). They are found on the regions

of America (11%), Europe (24%), East Mediterranean (6%), Western Pacific (48%), Africa (3%) and Southeast Asia (8%) (WHO 2012). It was also found that from year 2000 to 2010, the global severity of cigarette smoking by male teenagers increased over 100 times with an average of 865 pieces of cigarettes or 43 packs per day worldwide.

In Malaysia, a ten-year survey was conducted by a global organisation called Global Adult Tobacco Survey (GATS 2012) and found that 43.9% of cigarette smokers

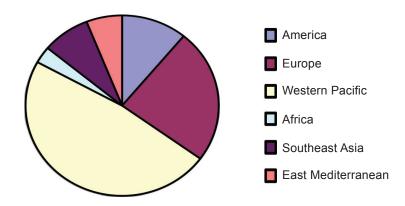


Figure 2. Global illustration from six regions where cigarettes are smoked for their whole life from a total population of 43 trillion.

were male teenagers. Active teenage cigarette smokers in Malaysia smoke on a daily basis, as surveyed by GATS (2012), are local citizens, transient visitors from foreign countries, regular male teenage tourists and children of foreign immigrants.

Another study was done by Lim *et al.* (2010) for 1180 teenage students in comparison and in contrast with the GATS (2012) survey, with a response rate of 94.7% (1117). Of the 1117 respondents, 705 (63.1%) are former smokers and 397 (35.5%) are current smokers. Among the current smokers, 36 (9.1%) was daily smokers, 48 (12.1%) smoke once every two days, 131 (33.0%) smoke once or twice a week and the rest smoke once a week (Lim *et al.*, 2010; WHO-MOH 2012).

The GATS survey in comparison with the studies done by Lim *et al.* (2010) was found to be higher as prevalent in male adolescents' cigarette smoking. It was also higher compared with what is reported by the MOH (2006) spanning from the city of Kota Bharu in the state of Kelantan to the city of Petaling Jaya in the state of Selangor (Afiah *et al.* 2006).

The prevalence of cigarette smoking in Malaysia, complicated by gender, race, ethnicity and culture, particularly influenced male teenage cigarette smokers who continue the chain of influence among their peers (Wakefield *et al.* 2000; Naing *et al.* 2004; Poland *et al.* 2006).

DISCUSSION

Influence is a determinant of health that increases the prevalence of cigarette smoking among male teenagers (Child & Wi 2010; Ferrante *et al.* 2010; Watsen *et al.* 2010).

Teenage boys are perceived to influence their peers of the same gender (Lim *et al.* 2010; Afiah 2006; Ferrante *et al.* 2010).

The perception of a relief from stress after so much of studying and thinking makes teenage boys influence each other (Isohanni *et al.* 2006; Child *et al.* 2010; Jeanfreau *et al.* 2010; Lee *et al.* 2005).

Aside from gender, the successful lifestyle coming from a specific race, ethnicity and culture are also influential (Isohanni *et al.* 2006; Gonseth *et al.* 2012; Poland *et al.* 2006). Eventually these become influential to young boys making them actively smoke cigarettes as a part of thier lifestyle.

Lastly, cigarettes used for entertainment influences male teenagers to actively smoke cigarette smoking (Isohanni *et al.* 2006; Afiah *et al.* 2006). According to Poland *et al.* (2006), active cigarette smoking entertains the social appetite and social life. It is also entertaining when young boys in Malaysia tend to show off their ability to buy cigarettes to make their social status higher as influenced by adults (Lim *et al.* 2010; Naing *et al.* 2004).

These health determinants were evaluated using a system called health screening. Health screening is done in public and private schools in Malaysia by GATS (2012) and MOH (2007). High school young boys ages 13–19 years old were interviewed with survey forms to know their perceptions regarding smoking cigarettes (WHO-MOH 2012).

Health screening also involved organisations to visit hospitals and to analyse statistics on average teenage adolescents confinement taken yearly with collaborations from the MOH (Gabing 2010). A schedule of yearly statistical presentation among other healthcare institutions and hospitals gather together in a certain place to present findings or analyse statistical data collected from hospital confinement, mortality and discharged patients with presenting illnesses and diseases as caused by cigarette smoking (MOH 2007). Another form of health screening is being done annually on a continuous research conducted by Disease Control Division (MOH 2007).

The survey results found that active cigarette smokers among teenage boys are divided into current smokers, daily smokers, occasional heavy smokers and former daily (not more than 5 years ago) smokers (Lim *et al.* 2010; GATS 2012; MOH 2007). This impacts the MOH's healthcare delivery system since teenagers' health are at risk if they are influenced to become active cigarette smokers (Lim *et al.* 2010; WHO 2008; Lee *et al.* 2005). There is a need to ensure that the Malaysian government invests strategies to control or decrease the number of teenagers who smoke (WHO-MOH 2012).

Implications to Health Promotions

WHO-MOH (2012) implemented varieties of programmes to control cigarette smoking with an aim to reduce its consumption.

One of its programmes is to promote pictorial health warnings on cigarette packs with a general message that says *Smoking Causes Harm to Health* (MOH 2008). These photos were further enhanced when other researchers suggested showing pictures of cancerous organs i.e. throat, neck, lung, mouth and brain cancers (Azam & Maizura 2004). Another illustration of these pictures suggested by Azam and Maizura (2004) used heart failures with ischemic tissues, chronic obstructive pulmonary diseases, veins with plaques as adopted from anatomy and physiology books (Tortora & Derrickson 2010).

Another programme implemented by the Malaysian MOH (2007/2008) launched a campaign called *Say No* in 2004. This campaign was first initiated by the Southeast Asian Tobacco Control Alliance and was soon adopted by the Malaysians in 2008. The MOH used teenagers to rally and march across main roads and all over national highways of Malaysia carrying a banner with printed words saying *Tak nak* (Say No) (MOH 2008). This kind of campaign may stimulate the minds of influenced teenagers that it is not good to smoke (MacFarlane *et al.* 2010) and that egos can be boosted in other ways that are non-health hazard related (MOH 2008).

Another campaign implemented by the Malaysian MOH (2008/2006) was adopted from the policies of the World Bank (Lewit *et al.* 1981) recommending governments worldwide to introduce a dedicated tax on tobacco products. Teenagers, as anticipated, cannot afford to buy cigarettes in their youth (Wakefield *et al.* 2000). However, some of the school age students in Malaysia who cannot afford a pack of cigarettes will buy a single stick (Lim *et al.* 2010), that is allowed by some states like Sabah and Sarawak (MOH 2006).

The control of the habit of cigarette smoking was not strong enough using taxes, that was why another policy to make the strategy stronger was initiated.

The Malayasian MOH (2006/2007) banned certain areas in Malaysia from smoking and used signboards with written warnings for smokers such as smoke-free area. The private and government owned institutions in Malaysia supported this strategy by imposing a fine on smokers caught by security personnel smoking within the 'no smoking' zone. Over 80% of institutions in Malaysia supported this campaign using a total cigarette smoking ban indoors with air-conditioners such as restaurants, hospitals and educational institutions (MOH 2006/2007). This is also practiced worldwide as indoorprivate institutions placed a smoking area that is located in a small location so that lesser people will smoke (Wakefield et al. 2000).

Analysis

The identified strategies on health promotions and illness preventions against active cigarette smoking acquired a critical understanding and demonstrated a commitment to apply professional values in complex healthcare situations. Active cigarette smoking in a Malaysia is a contemporary healthcare issue that needs to be identified.

This narrative review is for future reference among healthcare professionals to be vigilant in preventing illnesses or promoting *measures and* procedures to improve a person's wellbeing (Olla 2006; Mohide 1988).

The target of the Malaysian Millennium Development Goal (MMDG) (WHO-MOH 2012) in the health sector is to curtial the threats of cigarette smoking that impacts on teenage boys in Malaysia. Teenage active cigarette smokers influence other teenagers to smoke cigarettes (Lim *et al.* 2010; Watsen *et al.* 2010).

United Nations Development Programmes (UNDP) (2011) recommended to healthcare professionals a multi-disciplinary approach to encourarge individuals to join campaigns against cigarette smoking (WHO 2012) using healthcare initiatives.

Campaigns must endeavour to curtail chain smoking as a health hazard (Isohanni *et al.* 2010; Ferrante *et al.* 2010). Chain smokers among male teenagers can acquire diseases and illnesses that are morbid and very difficult to cure when they grow older (Kiechl *et al.* 2002; Tortora & Derrickson 2010).

This is the wider health threat that impacts teenage boys who smokes cigarettes (Lim *et al.* 2010) especially if their smokes affects their family members or peers who are confirmed to be pregnant or ill — extending to the severity of second-hand smoking.

CONCLUSION

The prevalence of active and second-hand cigarette smoking among male teenagers in Malaysia should be prevented.

It is therefore concluded that in order for healthcare initiatives, such as MMDG and UNDP, to overcome problems of cigarette smoking, a good strategy is important especially on campaigning preventive measures against the threats of chain cigarette smoking. In addition, an awareness campaign should emphasise on preventing teenagers' influential capabilities to non-cigarette smokers.

Active and second-hand cigarette smoking can cause chronic and/or incurable physiologic imbalances. That is why prevention is better than cure.

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Forecsting of Hydrological Time Series Data with Lag-one Markov Chain Model

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Planning and operation are important elements in water resource management. Rainfall forecasting is one of the conducts commonly used to extend the lead-time for catchments with short response time. However, it is difficult to obtain a high degree of accuracy in rainfall forecasting using deterministic models. Therefore, a probability-based rainfall forecasting model, based on Markov Chain provided a better alternative due to its ability to preserve the basic statistical properties of the original series. This method was especially useful in the absence of long-term recorded data, a rampant phenomenon in Malaysia. Comparison of statistics in the generated synthetic rainfall data against those of the observed data revealed that reasonable levels of acceptability were achieved.

Key words: Rainfall forecasting; Lag-one Markov chain; model; stochastic; synthetic rainfall data

Malaysia's climate is overwhelmingly characterised by uniform temperature, high humidity, copious rainfall and light winds. As in any parts of equatorial doldrums, intermittent rain and sunshine within a day is a norm, as such, a long period of clear sky is rare. One of the commonly identified problems in water resource management in Malaysia is unavailability of long-term historical records. Where there is data availability, it was found to be discontinuous. Since forecasting using rainfall data requires long-term continuous recorded historical data, a stochastic type of model simulation that can cope with these situations is proposed.

MODEL CONCEPTALISATION

In this study, the available hydrological records used were usually less than 100 years. In fact most of the records are less than 25 years. Even in the case of the longest record, the most extreme event such as drought or flood can be very different in magnitude with the next most extreme event. It is often debatable whether the extreme event is representative of the period recorded. The severity of a long drought can change drastically by adding or subtracting one year of its duration. To enable estimates of likelihood of severe events to be made, stochastic process is simulated where long sequences of events are generated. If the generation is done correctly, the hypothetical sequences would have as equal likelihood of occurrences in the future as in the observed records.

Any hydrological time series data are typically supported by two contributing factors namely random and persistence (stochastically deterministic) factor. Rainfall is regarded as the most basic weather variable, independent of temperature and evaporation. Therefore, generation of long-term synthetic rainfall data can provide basic sets of weather variable for

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long-term forecasting. In Lag-One Markov Chain Model, one-year historical data can be used to forecast the subsequent year's rainfall data. Similarly if a three-year historical data are used, we can forecast the next three-year rainfall data.

Daily rainfall data used in this model was available from three types of recordings namely the manual, automatic and data logger. Rainfall data from chart and data logger recordings were selected based on the period of data provided by the recorder. Data from either the data logger or manual method were selected to replace the non-available data in the chart recorder method. If all these three methods failed to supply the data for any particular day then the data from the nearby rainfall stations were used.

The time series rainfall data used in this study must satisfy certain requirements. The aim of the analysis is to find scale-independent properties. Thus, the series should span over a large range of scale. This means that the data has to be continuous and of high temporal and intensity resolution. All intensity levels must be correctly represented in the data. However this is difficult to be accomplished when measuring at high intensity resolution. It has been shown that an insufficient intensity resolution, which leads to erroneous representation of especially low-intensity rainfall, might attract artificial breaks in scaling behaviour.

In this study, analysis was made on daily rainfall data from 1974 to 2003 for eight rainfall stations in the Gombak river catchment areas in Selangor (one of the states in Malaysia). The Gombak river is geographically located at latitude 3° 8' 53" north of the Equator and longitude 101° 41' 44" east of the Prime Meridian on the map of Kuala Lumpur. The river drains from the main river which is the Klang river. The Klang river drains an area of about 1200² km extending from the headwaters in steep mountain forests of the Main Range in Peninsular Malaysia, to the river mouth spanning over a total length of 120 km. The location map of the catchment areas is as shown in *Figure 1*.

Gombak river catchment area was selected because of the long period of data available. For automatic station, 28 years of data were recorded. For data logger stations, different period of recorded data was found as the loggers were originally installed on different dates. Detail information about the location, period of data recorded and type of recordings is as shown in *Table 1*. Types of rainfall gauge available at the study area and its capability are shown in *Table 2*.

The types of data used in this study are as follows:

- 1. Automatic Station 30 year period of daily rainfall data.
- 2. Data Logger Station Period of daily rainfall data will depend on the date of the instrument installed.

Analysis was performed on the total monthly rainfall figures (mm): maximum rainfall for every month (mm); minimum rainfall for every month (mm); mean of the rainfall for every month (η); skew of rainfall amount for every month (γ); standard deviation rainfall amount for every month (σ) and coefficient of variation (C_v).

Benson and Matalas (1967) and Solomon (1976), found that regionalized parameters are more suitable than single site parameters because regionalization reduced operational bias due to temporal and spatial variations inherent in historic sequences. Based on these findings, the use of catchment area's average rainfall data instead of rainfall data from individual stations was found to be more desirable in stochastic method of generating synthetic data. The usage of catchment area's average

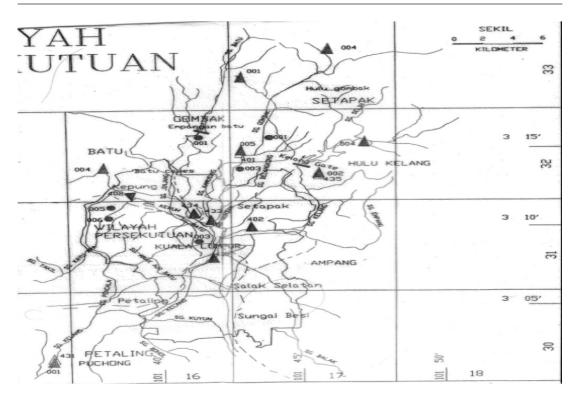


Figure 1. Location of rainfall stations at the Gombak river catchment area. (Source: Jabatan Pengairan dan Saliran (JPS), Malaysia, 2005).

| TABLE 1. Location, station number, period of data recorded and type of recording methods at |
|---|
| rainfall stations in the Gombak river catchment area. |

| Location | Station no. | Data period / recording method |
|---------------------------|-------------|---------------------------------------|
| P1 Genting Sempah | 3317004 | 1974–2002 / Automatic and data logger |
| P2 Air Terjun Sg. Batu | 3317001 | 1985–2003/ Automatic and data logger |
| P3 Kampung Sg. Sleh | 3217004 | 1993–2003/ Automatic and data logger |
| P4 Ibu Bekalan KM 16 | 3217001 | 1985–2003/ Automatic and data logger |
| P5 Kampung Sg. Tua | 3216001 | 1993-2003/ Automatic and data logger |
| P6 Gombak Simpang 3 | 3217005 | 1982–1999/ Automatic and data logger |
| P7 Empangan Genting Klang | 3217002 | 1993-2003/ Automatic and data logger |
| P8 Ibu Bekalan KM 11 | 3217003 | 1974–2002/ Automatic and data logger |

Table 2. Type of rainfall gauge and capability available at study area.

| Type of rainfall gauge | Instrument capability |
|------------------------|----------------------------|
| 1. Automatic | Monthly (Daily) / Annually |
| 2. Data logger | Monthly (Daily) / Annually |

rainfall data allowed better approximations of rainfall stochastic properties. The catchment's average rainfall is computed using Thiessen Polygon Method (Thiessen 1911). *Figure 2* shows a sample of Thiessen average rainfall data determination.

METHODOLOGY

Baki (1997) adopted the approach used by Adamowski and Smith (1972) by using runoff generation type model to generate daily rainfall data. A first order Markov model (also known as the Lag-one Markov Chain) was used to generate standardized daily rainfall data. The outline of model operations was as follows:

- 1. The daily recorded data was calculated: mean $(\overline{\chi_i})$ and standard deviation (σ_i) of everyday (*i*) in a year.
- 2. The overall serial correlation (r_i) of the recorded data was also calculated.
- 3. The standardized daily rainfall (Z_i) was computed.
- Normally distributed random numbers (t_i) with zero mean and unit variance are generated.
- 5. All negative daily rainfall values were set to zero.

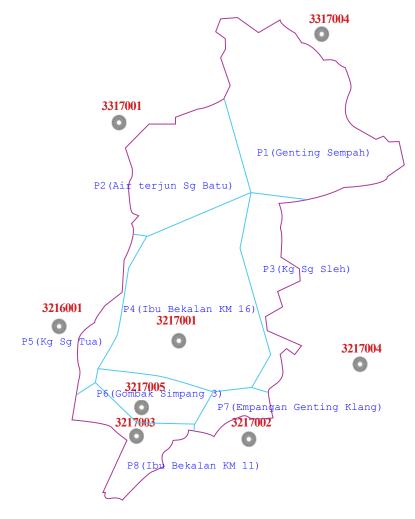


Figure 2. Average rainfall in the catchment areas computed using Thiessen (1911).

DATA ANALYSIS AND RESULTS

The data generated was analyzed using statistical components. *Table 3* shows the comparison of statistical parameters obtained from both recorded and generated data. It was found that the daily means were modeled satisfactorily. The daily standard deviations achieved were almost reasonable.

| Table 3. Comparison of statistics on recorded |
|---|
| and generated data. |

| Element | Mean | Std. deviation | Skew |
|----------------|-------|----------------|-------|
| Recorded data | | 9.229 | 1.589 |
| Generated data | 5.797 | 2.721 | 0.469 |

The daily estimated statistical parameters obtained are shown graphically in *Figures 3* to *5*. *Figure 3* shows the comparison of mean values for both recorded and generated data. *Figure 4* shows the comparison of standard deviation values for both recorded and generated data. *Figure 5* shows the comparison of skew values for both recorded and generated data.

DISCUSSION

In the validation of synthetic daily rainfall data generated from the proposed Lag-one Markov Chain model, three statistical parameters were analyzed namely mean, standard deviation and

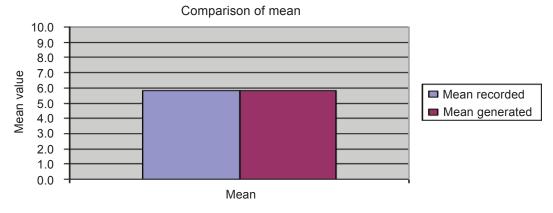
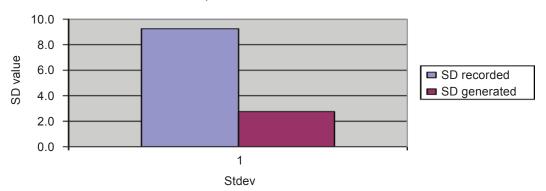


Figure 3. The Mean comparison of recorded and generated data.



Comparison of standard deviation

Figure 4. The standard deviation comparison of recorded and generated data.

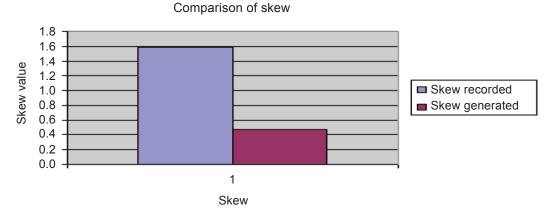


Figure 5. The Skew comparison of recorded and generated data.

skew values. The daily mean values obtained from generated data were found to be similar to the mean values of recorded data. It was also found that the model generateds reasonable data sequences of wet and dry days. However, standard deviation values of generated data were found to be lower than standard deviation values of recorded data.

The negative rainfall values presented in the historical rainfall records indicated missing rainfall data. Setting the negative rainfall values to zero affected the skew values produced by the proposed model. The skew values of generated data were found to be much lower than the skew values of recorded data. The differences of these values indicated that the generated data was almost normally distributed.

In generating synthetic daily rainfall data, *Fortran 90* could not detect the presence of leap years which occurred in the 26 years of rainfall records analyzed. By assuming that each year in the 26 years of rainfall record was a leap year i.e. every year has 366 days, *Fortran 90* produced additional numbers of generated data. Nevertheless, due to its small amount, the effect of these additional numbers of generated data were not significant and could be neglected.

CONCLUSION

The synthetic daily rainfall data forecasted from Lag-one Markov Chain model gave an approximation of the statistical properties on the historical record available. The statistics of the generated data seemed to be sensible in generating reasonable values of daily rainfall data, monthly and annual mean values, daily maximum values of rainfall data, monthly maximum and minimum values of rainfall data, as well as the length of sequences for both dry and wet days. Thus, the proposed model was able to give a quick analysis of daily rainfall data in stochastic hydrology.

APPLICATION AND RECOMMENDATION FOR FURTHER STUDY

Daily rainfall data analysis using the proposed method could be useful for agricultural planning in Malaysia, as the method enables the planners to regionalize the characteristics of wet and dry days. The proposed method could also be potentially applied as an early warning system of probable natural disaster. The model could also be tested on other climatic data such as temperature and wind.

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Assessment Attributes on Effective Construction Management for Property Developers in Malaysia

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Property developers are financially capable of running construction projects. Nevertheless, in Malaysia it was found that many of these construction practitioners failed to provide effective construction management which affected society. The success of a project and its Critical Success Factors (CSFs) are considered as tools to improve the effectiveness of project management. Even though many researchers have conducted studies on CSFs, the use of CSFs has remained ambiguous. The main objective of this study is to validate the proposed assessment attributes obtained from the property developers based on CSFs. A questionnaire survey was conducted to validate this predetermined attributes. Comparison was made with attributes from *Project Management Book of Knowledge* in order to explore the underlying suitability of the proposed attributes with the study area in terms of climate, economy, etc. Factor Analysis was adopted to investigate the group component relationships. This study focuses on the link between CSFs and the implementation of an effective construction management specifically for property developers in Malaysia.

Key words: Critical Success Factors; property developer; assessment; construction industry; Malaysia; management; factor analysis

Malaysia has shown very rapid growth in property and construction since the 1970s. The competition among property developers has created major changes in the construction industries mainly in the design and infrastructure, in order to satisfy the needs of the property buyers. Eventually, the capability of property developers varies. Project management, including the tools, techniques, and knowledge-based practices applied to manage the creation of products and services, is becoming an increasingly accepted and applied discipline across industry sectors (Jugdev et al. 2007). Adoption of project management is used as 'a method' for solving complex organizational problems. Such a viewpoint treats project management as one of the several ways of handling organizational activity. Similar arguments and standpoints are found in numerous project management research texts (Söderlund 2004).

Research on Critical Success Factors (CSFs) in the construction industries were mainly based on theoretical rather than empirical evidences (Khosrowshahi & Howes 2005). But, what is the extent of expectation that could be obtained from CSFs? The outcome of this study is therefore to determine the measurement arrays as attributes in developing an effective costruction management (CM) assessment for property developer in Malaysia.

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Literature Review

CSFs has been defined as a tool to identify executive information needs (Rockart 1982). Rockart et al. (1979) identified four prime sources of CSFs in any industry, which are: (1) Structure of the industry: has its own set of CSFs which are dependent on its characteristics; (2) Competitive strategy, industry position and geographic allocation: each organization has its own strategies and strategic plan due to the nature of the industry in which it operates; (3) Environmental factors: the effects of the environment upon the organization behaviour are essential to understand the CSFs; (4) Temporal factors: CSFs changes with the change of the organization priorities, where the areas of activity for success changes and some activities become more critical and others become less critical (Elwakil et al. 2009).

In Managing Information Systems, CSFs examine their existing methodologies, and from time to time, CSFs has been widely used by other industries, including the construction industry. In the construction industry, CSFs are integrated with eight elements that are used as benchmarking parameters which are: structure of industry; competitive strategy; market conditions; political environment; organizational structure; technical applications; employee enhancements and process benchmarking (Rockart 1982; Sanvido et al. 1992; Abraham 2003). Success is defined by Ashley et al. (1987) as 'results much better than expected or normally observed in terms of cost, schedule, quality, safety and participant satisfaction'. The investigation of success factors in construction industries have attracted the interest of many researchers and many studies have been conducted with the aim of providing valuable insights into how to consistently achieve superior results for the projects. Although construction projects are by their nature repetitive activities, each one has its own characteristics and circumstances (Salleh 2009).

Chan (2004) identified five primary CSFs from 44 identified factors, which are: projectrelated factors; project procedures; project management actions; human-related factors and external environment (Yong & Mustaffa 2011; Doloi et al. 2011) established attributes that relate to schedule and performance, listing 55 attributes that were subsequently grouped into six CSFs and seven Critical Failure Factors. Those factors are project managers' competence, supportive owners, top management monitoring, feedback, and co-ordination. Love et al. (2002) identified 55 attributes and grouped them into five CSFs for public-private partnership projects in the United Kingdom. The five categories were: effective procurement; project implementation ability; government guarantees; favourable economic conditions, and the available financial market. Abraham (2004) identified seven CSFs that influence the success of construction industries which are: competitive strategy; market analysis; political environment; economic environment; technical application; employee/ organizational enhancement and process benchmarking. Saqib (2008) listed the top five CSFs affecting the construction industries in Pakistan, developed from 77 identified factors which are: contractor-related factors; project management factors; procurement-related factors and design team-related factors. Marc Hockins (Stolton & Leverington 2006) proved that CSFs are the best methodology to develop an executive monitoring system to contain corporate-wide indicators of success (Elwakil et al. 2009). In this study, the function of CSFs is reversed by using attributes obtained from the questionnaire survey conducted.

RESEARCH METHODOLOGY

In order to achieve the objective of this study, a questionnaire survey was distributed among the practitioners in the construction industry which included government sectors, consultants, property developers, contractors and others (suppliers, manufacturers, planners and others.). The questionnaire contained 37 nominated success factors for property developers. It was developed from an extensive literature review and was consolidated by a series of pilot studies conducted in several states of Malaysia.

Five hundred sets of questionnaires were distributed within Malaysia. The distribution was categorized into few regions which consisted of the central region (Selangor and Kuala Lumpur), northern region ((Pulau Pinang, Kedah, Perak and Perlis), eastern region (Kelantan, Terengganu and Pahang), southern region (Negeri Sembilan, Melaka and Johor) and East Malaysia (Sabah and Sarawak). Data were collected and analyzed using Factor Analysis in statistics via SPSS (V.20). The Factor Analysis technique used was Principal Component Analysis (PCA) where effective variables are used to identify the principal factors. This techniques enables a more in-depth understanding of factor grouping techniques to underpin the success measures (Robinson et al. 2005). PCA can also be used for hypothesis testing or in searching for constructs within a group of variables (Sommerville et al. 2004). It is a series of methods for finding clusters of related variables and hence an ideal technique for reducing a large number of items into a more easily understood framework (Norusis 2008). Since the numbers of variables for CSFs for pre-determined attributes were about 37 numbers, Factor Analysis was used in this study, to converge these numbers to make it more reliable.

In order to determine the validity of the questionnaire developed in this study, Cronbach's alpha was tested to provide an accurate estimate of internal consistency and indicates how well the items in the set were correlated to one another (Brown & Adams 2000). The internal consistency ranges between zero and one. A commonly-accepted rule of thumb is that scores of above 0.70 are considered acceptable (Nunnally 2010). In this study, Cronbach's alpha was computed at 0.791 which indicated that the items were in the form of a scale with reasonable internal consistency reliability.

DATA ANALYSIS AND RESULTS

Response Rate

A total of 344 questionnaires were satisfactorily completed, resulting to a total response rate of 68.8%. This is acceptable as according to Takim *et al.* (2004) and Peansupap *et al.* (2005); they stated normal response rate in the construction industries for postal questionnaires is approximately between 20% to 30%. The General Respondent Demographic showed that the majority of the respondents (48.3%) were from property developers as shown in *Table 1*.

The questionnaires were distributed to all practitioners in the construction industries in Malaysia. Based on *Table 1*, it was found that the highest respondents were property developers (48.4%), followed by contractors (20.6%), consultants (14.2%), the government sector (12.5%) and others (4.4%).

| | Type of organization | Frequency | Percent | Valid prcent | Cumulative percent |
|-------|----------------------|-----------|---------|--------------|--------------------|
| Valid | Government | 43 | 12.5 | 12.5 | 12.5 |
| | Consultants | 49 | 14.2 | 14.2 | 26.7 |
| | Property developer | 166 | 48.3 | 48.3 | 75.0 |
| | Contractor | 71 | 20.6 | 20.6 | 95.6 |
| | Others | 15 | 4.4 | 4.4 | 100.0 |
| | Total | 344 | 100.0 | 100.0 | |

Table 1. Respondent to questionnaire based on General Respondent Demographics.

Ranking of Critical Success Factors

The first analysis was performed to rank the nominated factors based on the mean values of the responses. In this study, it was assumed that if two or more factors happen to have the same mean values, then the one with the lowest standard deviation would be assigned as the highest important rank among the nominated factors. In addition, factors with means exceeding or equal to the value of four are recognized as CSFs based on the consensus of the respondents. In this study, 15 factors were identified as CSFs having significant influence on the success of this study. *Table 2* shows the ranking of these factors according to the value of their statistical means. The CSFs identified in this study were largely in line with the findings of other researchers in the field of CSFs. Nevertheless, unlike other studies on CSFs, this study led to the refinement of the assessment attributes that would ensure affective construction management in Malaysia.

Factor Analysis

In this study, Factor Analysis is used to explore and detect the underlying relationships among the identified CSFs. This statistical technique identifies a relatively small number of factors that can be used to represent relationships among sets of many interrelated variables. Various tests

| Success factors | Mean | Standard deviation | Ranking |
|---|------|--------------------|---------|
| Construction Manager's organizing skills | 4.60 | 0.644 | 1 |
| All aspects of safety, with particular reference to implementation of safety programme | 4.51 | 0.752 | 2 |
| Monitoring results of specific construction as per required with relevant quality standards. Identifying ways to eliminate causes of unsatisfactory | 4.43 | 0.551 | 3 |
| Evaluating overall construction performance on a regular basis to provide confidence that the constructions as per required quality standard | 4.41 | 0.646 | 4 |
| Identifying which quality standards are relevant to the construction and execution as per requirement | 4.40 | 0.654 | 5 |
| Identifying ways to eliminate causes of unsatisfactory | 4.35 | 0.769 | 6 |
| Construction Manager's experience and capabilities with particular reference to technical, administrative, human relations and communication skills | 4.30 | 0.768 | 7 |
| Construction Manager's leadership skills | 4.30 | 0.675 | 8 |
| Construction planning processes in design, implementation and monitoring are as per approved documentation. | 4.28 | 0.511 | 9 |
| Developing individual and group skills to enhance construction performance. | 4.27 | 0.607 | 10 |
| Subdividing major construction deliverables into smaller, more manageable components | 4.25 | 0.667 | 11 |
| Carrying out the Construction plan by performing the activities included there in | 4.22 | 0.618 | 12 |

Table 2. Ranking of success factors based on 'Mean' value.

| Table 2 (Cont.). Kanking of success factors based on Mean Value. | | | | | | | |
|--|------|--------------------|---------|--|--|--|--|
| Success factors | Mean | Standard deviation | Ranking | | | | |
| Analyzing activity sequences | 4.18 | 0.656 | 13 | | | | |
| Estimation of period for each construction stage | 4.17 | 0.611 | 14 | | | | |
| Company have their own monetary capabilities before start work | 4.17 | 0.556 | 15 | | | | |
| Coordinating changes across the entire construction | 4.15 | 0.763 | 16 | | | | |
| Establish a written scope statement as basis for future construction decisions | 4.14 | 0.687 | 17 | | | | |
| All aspects of safety, with particular reference to safety monitoring programme | 4.13 | 0.759 | 18 | | | | |
| Construction Manager's commitment to meeting cost, schedule, safety and quality commitment | 4.13 | 0.836 | 19 | | | | |
| Defining enhancement steps for opportunities and responses to threats | 4.12 | 1.029 | 20 | | | | |
| All aspects of safety, with particular reference to safety programme regulations and requirement | 4.12 | 0.655 | 21 | | | | |
| Evaluating risks to assess the range of possible construction outcomes | 4.11 | 0.944 | 22 | | | | |
| Analyzing activity durations | 4.08 | 0.767 | 23 | | | | |
| Construction Manager's co-ordinating ability and rapport with owner/owners representatives | 4.07 | 0.869 | 24 | | | | |
| Construction Manager's co-ordinating ability and rapport with contractors/subcontractors | 4.07 | 0.729 | 25 | | | | |
| Committed to disburse effective construction process to the organization throughout the project | 4.07 | 0.642 | 26 | | | | |
| Evaluating risks interactions to assess the range of possible construction outcomes | 4.05 | 0.918 | 27 | | | | |
| Developing an approximation (estimate) for the costs of resources | 4.04 | 0.729 | 28 | | | | |
| Analyzing activity requirements | 3.97 | 0.799 | 29 | | | | |
| Controlling changes to the construction budget | 3.93 | 0.777 | 30 | | | | |
| Allocation of overall cost estimation to individual work items | 3.92 | 0.632 | 31 | | | | |
| Responding to changes in risk over the course of the construction | 3.90 | 1.091 | 32 | | | | |
| Developing an approximation (estimate) for the costs of resources | 3.89 | 1.010 | 33 | | | | |
| Determining which risks are likely to affect the construction and documenting the characteristics of each | 3.88 | 0.986 | 34 | | | | |
| Determining resources (people, equipment, materials) | 3.75 | 1.058 | 35 | | | | |
| Monitoring changes to the construction schedule | 3.73 | 0.998 | 36 | | | | |
| Determining quantities | 3.60 | 0.973 | 37 | | | | |

Table 2 (Cont.). Ranking of success factors based on 'Mean' value.

are required for the appropriateness of this method for factor extraction.

In this study, 37 numbers of CSFs were obtained as shown in *Table 3* subjected to Factor Analysis using PCA and varimax rotation. PCA is a common method in Factor Analysis. It involves the generation of linear combinations of variables in Factor Analysis so that the variance present in the collected data are considered. This analysis summarizes the variability in the observed data by means of a series of linear combination of 'factors'. Each factor can be viewed as a 'supervariable' comprising a specific combination of the actual variables examined in the survey. The advantage of this method over other factor analytical approaches is that the mathematical representation of the derived linear combinations avoids the need for the use of questionable causal models (Johnson & Carter 1993; Shen & Liu 2003).

Interpretations of the Components Group

In this study, eight numbers of group components were extracted using varimax rotation Factor Analysis. In accordance to Burgees (2006), based on Factor Analysis output for factor loading the results on all attributes could be defined as very high (0.6), high (0.3), and ignored (less than 0.3) (Kozak-Holland & Procter 2013).

| | 5 | | 0 1 | 1 | | | | |
|---|------|---|-----|----------|--------|----|---|---|
| | | | C | Group co | ompone | nt | | |
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| Evaluating risks interactions to assess the range of possible construction outcomes | .957 | | | | | | | |
| Defining enhancement steps for opportunities and responses to threats | .903 | | | | | | | |
| Determining resources (people, equipment, materials) | .866 | | | | | | | |
| Determining which risks are likely to affect the construction and documenting the characteristics of each | .822 | | | | | | | |
| Evaluating risks to assess the range of possible construction outcomes | .789 | | | | | | | |
| Monitoring changes to the construction schedule | .760 | | | | | | | |
| Developing an approximation (estimate) for the costs of resources | .739 | | | | | | | |
| Determining quantities | .637 | | | | | | | |
| Developing an approximation (estimate) for the costs of resources | 409 | | | | | | | |
| Construction Manager's organizing skills | .021 | | | | | | | |
| Subdividing major construction deliverables into smaller, more manageable components | 008 | | | | | | | |

Table 3. Factor analysis at each group component

| | Group component | | | | | | | |
|--|-----------------|------|------|------|------|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| Construction planning processes in design, implementation and monitoring are as per approved documentation | | .677 | | | | | | |
| Establish a written scope statement as basis for future construction decisions | | .619 | | | | | | |
| Analysing activity sequences | | .582 | | | | | | |
| Construction Manager's leadership skills | | .494 | | | | | | |
| Construction Manager's experience and capabilities with particular reference to technical, administrative, human relations and communication skills | | .107 | | | | | | |
| All aspects of safety, with particular reference to safety monitoring programme | | 095 | | | | | | |
| Evaluating overall construction performance on a regular basis to provide confidence that the constructions as per required quality standard | | | .636 | | | | | |
| Identifying which quality standards are relevant to the construction and execution as per requirement | | | .553 | | | | | |
| Analysing activity requirements | | | .280 | | | | | |
| Co-ordinating changes across the entire construction | | | .037 | | | | | |
| Company have their own monetary capabilities before start work | | | | .685 | | | | |
| Developing individual and group skills to enhance construction performance | | | | .484 | | | | |
| Construction Manager's co-ordinating ability and rapport with contractors/ subcontractors | | | | .139 | | | | |
| Analysing activity durations | | | | | .715 | | | |
| Controlling changes to the construction budget | | | | | 666 | | | |
| All aspects of safety, with particular reference to implementation of safety programme | | | | | .245 | | | |
| Identifying ways to eliminate causes of unsatisfactory | | | | | 156 | | | |

| Table 3 (Cont.). | Factor analysis at | t each group component |
|------------------|--------------------|------------------------|
| | | |

| | | | C | broup co | ompone | ent | | |
|---|---|---|---|----------|--------|------|------|------|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| All aspects of safety, with particular reference to safety program regulations and requirement | | | | | | .568 | | |
| Committed to disburse effective construction process to the organization throughout the project | | | | | | .535 | | |
| Construction Manager's commitment to meeting cost, schedule, safety and quality commitment | | | | | | .060 | | |
| Monitoring results of specific construction as per required with relevant quality standards. Identifying ways to eliminate causes of unsatisfactory. | | | | | | | .765 | |
| Construction Manager's co-ordinating ability and rapport with owner/owners representatives | | | | | | | .480 | |
| Allocation of overall cost estimation to individual work items. | | | | | | | .148 | |
| Responding to changes in risk over the course of the construction | | | | | | | .119 | |
| Carrying out the Construction plan by performing the activities included there in. | | | | | | | .069 | |
| Estimation of period for each construction stage | | | | | | | | .096 |

Table 3 (Cont.). Factor analysis at each group component

Group Component 1: Construction Integration Management (CIM). This includes the processes required to ensure that the various elements of the project are properly co-ordinated. It involves trade-off among competing objectives and alternatives in order to meet or exceed stakeholders' needs and expectations. In this study, nine attributes were integrated in this group component as listed below:

- 1. Evaluating risks interactions to assess the range of possible construction outcomes
- 2. Defining enhancement steps for opportunities and responses to threats

- 3. Determining resources (people, equipment, materials).
- 4. Determining which risks are likely to affect the construction industry and documenting characteristics
- 5. Evaluating risks to assess the range of possible construction outcomes
- 6. Monitoring changes to the construction schedule
- 7. Developing an approximation (estimate) for the costs of resources
- 8. Determining quantities; and
- 9. Developing an approximation (estimate) for the costs of resources.

Group Component 2: Construction Scope Management (CSM). This includes the processes required to ensure that the project includes all the work required, in order to complete the project successfully. It is primarily concerned with defining and controlling what is not included in the project. In this study, four attributes were integrated in this group component as listed below:

- 1. Construction planning processes in design, implementation and monitoring are as per approved documentations
- 2. Establish a written scope statement as basis for future construction decisions
- 3. Analyzing activity sequences; and
- 4. Construction manager's leadership skills.

Group Component 3: Construction Quality Management (CQM). This includes the processes required to ensure that the project will satisfy the need for which it is undertaken. It includes all activities on the overall management function that determine the quality policy, objectives, responsibilities and its implementation within the quality system. In this study, two attributes were integrated in this Group Component as listed below:

- 1. Evaluating overall construction performance on a regular basis to provide confidence that the construction is as per required quality standard; and
- 2. Identifying which quality standards are relevant to the construction and execution as per requirement.

Group Component 4: Construction Coordination Management (CCM). It includes the processes that ensure cost and human component occupancy will satisfy the necessity of contractors/subcontractors competencies. This relates to progress report, progress payment and others. Attributes under this Group Component are:

1. Companies have their own monetary capabilities before the start of work

- 2. Developing individual and group skills to enhance construction performance; and
- 3. The ability of the Construction manager's in co-ordinating rapport with contractors and subcontractors.

Group Component 5: Construction Discrepancies Management (CDM). It relates to managing discrepancies and changes that occur during construction period such as construction activities, budget, time and others. In this study, two attributes were extracted into this Group Component as follows:

- 1. Analyzing activity durations; and
- 2. Controlling changes to the construction budget.

Group Component 6: Construction Process Management (CPM). This refers to the ensemble of activities in planning and monitoring of the process performance. In this study, two attributes were integrated in this Group Component as follows:

- 1. All aspects of safety, particularly on regulations and requirements of safety programe; and
- 2. Commitment to disburse effective construction process to the organization through the project.

Group Component 7: Construction Deliverability Management (CDM). It refers to the capability of the company to produce the end products as per expectation. In this study, two attributes were extracted in this Group Component as follows:

- Monitoring results of specific construction trade as per relevant quality standards. Identifying ways to eliminate causes of unsatisfactory products; and
- 2. The ability of Construction Manager in co-ordinating rapport with client and its representatives.

CONCLUSION

This study identified and analyzed the possible assessment attributes on effective CM for property developers in Malaysia. Identification of CSFs were used as measurement tools to determine its effectiveness. The findings of this study were generally in line with the earlier studies performed on CSFs which have been established by other articles in journals. Nevertheless, the findings of this study further enforced the results obtained from CSFs analysis and established the assessment attributes.

In this study, Factors Analysis was used to converge the 37 numbers of identified CSFs into eight groups. Out of the 37 attributes established in this study, 13 were eliminated due to the result obtained from Factor Analysis which was below 0.3 (less impact). It was found that CIM was the highest group component. For future work, the correlation rank at each element of the group components will be further investigated and defined using the Structural Equation Method.

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