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# Effect of Vinyl Acetate Effluent in Reducing Heat of Hydration of Concrete

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#### Abstract

Waste generation especially in surface coating industries is a potential pollutant to the environment globally. Waste disposal in the form of recycling or reused has gained some recognition as a raw material in many kind of interested fields. This study is therefore, aimed at utilizing Vinyl acetate effluent generated from manufacturing process of paint as polymer modifier in concrete. The research highlights the influence of Vinyl acetate effluent on setting time and control of heat of hydration in concrete. Three specimens containing 0%, 2.5% and 5% of Vinyl acetate effluent by weight of cement were prepared in cubic moulds. The temperature rises due to heat of hydration in all mixes were recorded. Finding shows that incorporating Vinyl acetate effluent considerably delayed the setting time in cement paste and reduced the total temperature rise in polymer modified concrete. Therefore, addition of Vinyl acetate effluent polymer in concrete may improve the properties of concrete to some extent particularly in mass concrete production. Keywords: heat of hydration, vinyl acetate effluents, polymer modified concrete, setting time, waste disposal

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# 1. Introduction

Products of paint and coating industries have become globally demanded item. Wide application of paint and coating made the production of these materials to increase tremendously. One of the major paint industries that is growing steadily is the water based paint. The increase of this type of paints becomes essential due to the environmental regulations and health concerns of the solvents used. In paint production, polyvinyl acetate is commonly used. These polymers compared to others are extensively used to produce emulsion paints for interior application, because the resulting paint are most durable, flexible, adhere strongly, dry quickly and colorless (Randall, 1992).

There is progressive increase in the production of this paint to match the increase demand for the paint. However, the production of this paint is characterized with generation of waste in form of Vinyl acetate effluents. Increase in production therefore, directly influences the volume of waste generated thereby posing waste disposal problem. For instance, paint consumption in Malaysia is expected to grow by about 3.5% per annum, and is

forecasted to about 166,000 metrics tones in 2014 (Reg, 2010). This may contribute to the increase of waste generation and landfill occupation by these industries. This figure has a tendency to increase with advancement in economic activities. Any means of recycling this waste would result to economic advantage as well as solved environmental pollution problem.

Portland cement as a binding agent is a well-known building material occupying indispensable place in construction work, which reacts when it comes into contact with water (Ghambir, 2004). The hydration of Portland cement is an exothermic chemical reaction and as such heat is produced during hydration phases. The reaction is known to be influenced by constituents of the binding systems (Neville, 1995). The development of heat during the setting and hardening phases can be high in mass of concrete such as dams, bridge deck concrete, tunnel lining etc. The phenomenon may also occur in structure constructed during inclement weather. In mass concrete for instance during hardening, heat loss should be control so that the temperature stresses do not exceed the tensile strength develop by the concrete. These may be achieved by removing the excess heat through pre-

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cooling of the material or the use of low heat cement. However, the use of some material could have the effect of reducing the heat generated during hydration reaction. Inclusion of pozzolanic material and chemical admixtures (retarders and superplasticizers) have influence on reduction of heat of hydration (Abdul Awal and Warid Hussin, 2011; Atis, 2002; Łaćniewska-Piekarczyk, 2013; Siddique and Bennacer, 2012).

There is growing interest to recycle waste material from paint production by using it to modify concrete mixes. Several works have been done in the area of adding and replacing the cement with the paint waste to improve mechanical strength and durability of concrete (Almesfer *et al.*, 2012; Ismail and Al-Hashmi, 2011; Nehdi and Sumner, 2003; Ismail *et al.*, 2011; Noruzman *et al.*, 2013). However, there is less information about the effect of Vinyl acetate effluent on reducing heat of hydration in concrete. The application of this waste could delay the hydration process of cement composite hence, may have positive effect on reducing heat of hydration. This study is aimed at characterizing the influence of the Vinyl acetate effluents on setting time of cement and heat of hydration of concrete.

# 2. Experimental Program

# 2.1 Materials

Ordinary Portland Cement (OPC) complying with BS EN 197-1: 2000 was used to prepare specimen for all test perform in this study. Table 1 presents the chemical compositions of the cement. The aggregates used include crushed granite and naturally occurring river-washed quartz sand with maximum nominal sizes of 10 mm and 2.75 mm respectively. The sand has fineness of 2.57 and tap water was used for mixing concrete.

# 2.2 Characteristics of Polymer Vinyl Acetate Effluents

Vinyl acetate effluents are typical polymer waste generated from the production of polymer dispersion factory located at the south of Malaysia. The wastes were collected from equipment cleaning activity which is in liquid form and milky white in color. The waste specimen had a pH value of 7.16 and total solids content is 42%.

# 2.2.1 Functional Group

Fourier Transform Infrared Spectrometer (FTIR) was used to identify chemical compounds in the specimens of the Vinyl acetate effluents. An IR spectrum was measured by scanning the specimen with continuous wave length from 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup>. Two sources point of effluents were taken and compared to the control (pure Vinyl acetate latex).

# 2.2.2 Chemical Compositions of Vinyl Acetate Effluents Specimen of Vinyl acetate was tested using Inductively Coupled

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	$SO_3$	MgO	LOI
20.1%	4.9%	2.4%	65%	2.3%	3.1%	0.85

Plasma Mass Spectrometry (ICP MS) to determine chemical compositions of the effluents. The instrument used was Perkin Elmer Sciex Elan 6100. High content of fine particles sample as BOD, COD, TSS, TDS were also measured in accordance with American Public Health Association (APHA) standard.

# 2.2.3 Scanning Microscopic Microscope (SEM)

Morphology characteristics of Vinyl acetate effluents were observed using high magnified Scanning Electron Microscope (SEM) Tabletop SEM Hitachi TM 3000. Specimen in liquid form was prepared for the test by drying in oven at 70°C for 4 hours and stored in the desiccator after removal from oven.

#### 2.3 Mix Proportion

The mix concrete was designed in associated with BS EN 206-1:2000 and tabulated in Table 2. Other two specimens were prepared by addition of 2.5% and 5% to the reference mix giving a total of three specimens with 0%, 2.5% and 5% of polymer by weight of cement. Concrete containing Vinyl acetate effluents were cured according to JIS A1171:2000. Meanwhile, the control specimens were cured as indicated in BS EN 12390-2: 2009.

# 2.4 Workability and Compressive Strength

Slump test was carried out following BS EN 12350-2:2009 to determine the workability of the concrete specimens. Slump values were taken in height (mm). Also, the compressive strength test was conducted in accordance with BS EN 12390-3:2009. Concrete were prepared with addition of Vinyl acetate effluent as well as control specimens in 100 mm sizes cube. Averages value of three specimens were tested at 7 and 28 days to represent compressive strength.

#### 2.5 Setting Time

The test was carried out to determine the time of setting of fresh hydraulics cementitious mixtures by means of the Vicat needle according to ASTM C191-04b. Cement pastes was mixed to normal consistency according to ASTM C187 and were prepared with 2.5% and 5% of Vinyl acetate effluents by weight of cement and compared to the control specimens. The penetration of the needle was taking at every 30 minutes until they were no noticeable penetration. The initial and final setting corresponds to the time when penetration is 25 mm and 0 mm respectively.

#### 2.6 Measurement of Heat of Hydration

The temperature rise due to heat of hydration of concrete mass

Table 2. Mix Design for Concrete

Constituents	PMC 0% VA	PMC 2.5% VA	PMC 5% VA
OPC (Kg/m <sup>3</sup> )	380	380	380
VA (Kg/m <sup>3</sup> )	0	9.5	19
Fine aggregate (Kg/m <sup>3</sup> )	824	824	824
Coarse Aggregate (Kg/m <sup>3</sup> )	1006	1006	1006
Water (Kg/m <sup>3</sup> )	209	204	198

was measured according to the method used in previous studies (Abdul Awal and Warid Hussin, 2011; Awal and Shehu, 2013; Mehmannavaz *et al.*, 2014). The method was used in place of calorimeter to compare the rise in temperature of the studied mixes. The heat of hydration evolved which is the sum of heat contained in the adiabatic system and heat losses to the environment. Assumed negligible heat losses, this heat contained in the system can be calculated using Eq. (1).

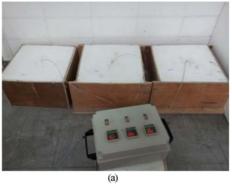
$$Q_t = C_c \Delta t \tag{1}$$

Qt = Specific of hydration (J/g)

$$Cc =$$
 Specific heat of concrete (known to be 0.880 J/g °C)

 $\Delta t$  = Temperature change in °C

A cubical plywood box 500 mm internally lined with 76 mm expanded polystyrene acting as insulator. Each batch of concrete was cast into the cubical box with internal dimensions of 300 mm. Thermocouple was inserted into the center of each box through drill hole of the polystyrene at the top surface and was connected to a SR1 temperature/humidity digital controller instrument to obtain temperature readings from each box (Fig. 1(a) and Fig. 1(b)). When concrete is poured into the box, heat was liberated by hydration process that subsequently increases the temperature of the concrete mass. The increase in temperature and subsequent drops was monitored with close interval during the first 24 hours and lesser frequency afterwards until the temperature dropped close to initial reading. The measurement continued up to 120 hours for all the mixes.



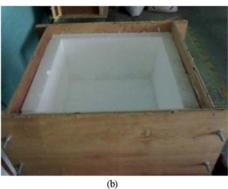


Fig. 1. (a) Arrangement of Cubical Plywood, (b) Insulator Plywood Box

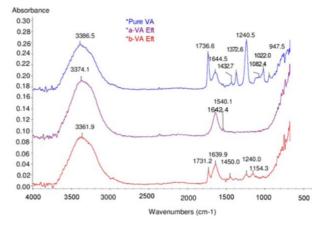


Fig. 2. Vinyl Acetate Effluents and Control Spectroscopy

# 3. Results and Discussion

### 3.1 Analysis of Spectrum

FTIR diagrams of Vinyl acetate effluents are shown in Fig. 2. Vinyl acetate is an organic compounds with the chemical structure of  $Ch_3COOCH=CH_2$  which are manufactured by free radical emulsion polymerization (Dunn and Beswick, 2002). However, in Table 3 shows the comparison between the effluents and the control (pure latex Vinyl acetate) which indicated the chemical structure of functional group. Specimen containing Vinyl acetate effluent (b) appears to be similar as control. Meanwhile, Vinyl acetate effluents specimen (a) present less bonding of chemical structure which is possibly caused by the effluent treatment process.

# 3.2 Characteristic of Waste Latex

Table 4 illustrates the characteristics of Vinyl acetate effluents used in concrete mixing. All parameter in the specimen are within the limits of standards effluents authorized by Department of Environment Malaysia (DOE, 2009), except a few parameters such as BOD, COD, TSS and mercury. These effluents falls under schedule waste generation that usually segregate the waste to disposal at landfill. Moreover, these effluents have higher contents of fine particles of the polymer product as a result of the equipment cleaning activities. Generally, the manufacture of latex paints consists of organic and inorganic pigments and

Table 3. The Absorption of Vinyl Acetate Effluents in cm<sup>-1</sup>

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Functional Group	VA latex (Pure)	VA Eft (a)	VA Eft (b)	Frequency Range
О-Н	3386.5	3374.1	3361.9	3400-3200
C=O	1736.6	-	1732.2	1750-1730
C=C	1644.5	1642.4	1639.9	1680-1600
CH <sub>3</sub>	1432.7, 1372.6	1504.1	1450	1450, 1375
С-О	1240		1240	1300-1000
C-O	1082.4		1154.3	1300-1000
С-О	1022			1300-1000
С-Н	947			1000-650

Parameter	Units	Standard B*	Tolerable Limit**	
pН	7.12	5.5-9.0	>5.0	
BOD	13363.00	50	-	
COD	77800.00	100	-	
TSS	8200.00	100	50000	
TDS	5460.00	-	-	
DO	2.72	-	-	
Temperature, °C	19.91	-	-	
Turbidity, NTU	23.64	-	-	
Tri-chromium (Cr <sup>3+</sup> )	0.1376	1	-	
Sulfate (SO <sub>4</sub> <sup>2–</sup> )	4514.00	-	3000	
Chloride (Cl <sup>-</sup> )	56.23	-	1000	
Nitrate (NO <sub>3</sub> <sup>2-</sup> )	27.53	-	500	
Sulphide (S <sup>2-</sup> )	< 0.002	0.5	-	
Cadmium (Cd)	0.0004	0.02	-	
Lead (Pb)	0.1302	0.5	100	
Phosphates (P <sub>2</sub> O <sub>5</sub> )	222.00	-	100	
Zinc (Zn)	1.0488	1	100	
Iron (Fe)	1.7051	5	-	
Calcium (Ca)	72.75	-	2000	
Magnesium (Mg)	9.7165	-	2000	
Sodium (Na)	1199.00	-	2000	
Copper (Cu)	2.6921	1	500	
Manganese (Mn)	0.9147	1	500	
Nickel (Ni)	0.0873	1	-	
Mercury (Hg)	3.4998	0.05	-	

Table 4. Physico-chemical Characteristics of the Vinyl Acetate Effluents (All values in mg/l except for pH, turbidity and temperature value)

\*\*Mandatory limits of chemical in concrete mixing according to BS EN 1008: 2002, ASTM C94, and (Mindness and Young, 1981).

dyestuff, extenders, cellulosic and non-cellulosic thickener, latexes, emulsifying agents, anti-forming agents, preservatives, solvents and coalescing agents. Due to the varying degree of chemicals used, the effluent contains appreciable concentrations of BOD, COD, suspended solids, toxic compounds and colors (Dey et al., 2004; Lorton, 1988; Randall, 1992). Furthermore, it also appears that most of the parameters are within the tolerable limit for mixing concrete except sulfate and phosphates ions shown slightly higher in percentage.

# 3.3 Morphology

Figure 3 shows the image of film formation of Vinyl acetate effluents. Film formation is associated with the evaporation of water from latex and time required to form a latex films depends on the characteristics of the dispersion, film thickness and environment condition. Once the continuous film is formed, it can undergo a subsequent aging process which can changes the properties of latex system (Dunn and Beswick, 2002). Energydispersive X-ray spectroscopy shows substances of 80.3% of carbon, 18.9% of oxygen, 0.3% of sodium and 0.42% sulphur in the specimen. The present of the sodium and sulphur in latex

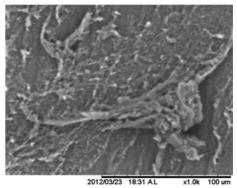


Fig. 3. SEM of Vinyl Acetate Effluents

show possibly the surfactant which are normally added to improve the stability of the properties of Vinyl acetate latex (Arnold et al., 2011).

# 3.4 Setting Time

Setting time cement is shown in Fig. 4. It is observed that incorporating of Vinyl acetate effluents in concrete slightly takes more time to set compared to the control specimens. The setting time increases with increase of the addition of polymer cement ratio. Initial setting cement for 0%, 2.5% and 5% were 131 min, 139 min and 150 min. Meanwhile, final setting cement was 210 min, 240 min and 270 min. However, all the specimens were satisfied as accordance in ASTM C150 which ideally initial setting time should not be less than 45 min and final setting time should not more than 375 min. This observation has similar results with others material (Jiménez et al., 2010). The setting time is delayed due to possibly action of the presence of the surfactant in the latexes which prevents the cement to completely hydrated (Ramakrishnan, 1992; Van Gemert, 2005). However, there is few mechanisms retardation effect that influences the behavior of organic particles in the cement component. As been reported by a recent research, these retardation is due to the interaction of polymer fine particles on the surface of the cement and also the changes of Ca(OH)<sub>2</sub> nuclei which delayed the formation of tricalcium silicate (C<sub>3</sub>S) to be hydrated. Besides that, the presence of the carboxylic acids and inorganic salts such as phosphates, zinc, lead as well as other impurities in the ingre-

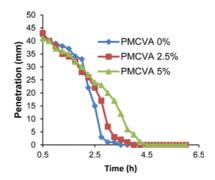


Fig. 4. Setting Time of Cement with Different % of Vinyl Acetate Effluents

dients may have some effect to the alteration of the hydration products (Khan and Muhammad-Ullah, 2004). Furthermore, the addition of more percentages of Vinyl acetate dosages to the cement paste may also delayed the setting time due to the larger specific area of particles sizes (Ray *et al.*, 1994; Denise A. Silva and Monteiro, 2006; Su *et al.*, 1991).

# 3.5 Workability and Compressive Strength Characteristics

As tabulated in Table 5, the value of slump increases with addition of polymer Vinyl acetate effluents, thus increase the workability of mixes. The increases are caused mainly by ball bearing action of polymer particles and entrained air as well as the dispersing effect of components in latex combines in water (Ramakrishnan, 1992). Meanwhile, the addition of 2.5% of Vinyl acetate effluents showed higher compressive strength compared to the control specimen. The increases are attributed to the sufficient amount of polymer particles that filled up the capillary pores, forming a closed packed layer on gel product surfaces on the unhydrated cement grains and developing silicate layers over the aggregates. As the water withdraws from hydration process or evaporates, the closed packed polymer particle coalesced into a continuous film to form a co-matrix intermingled with the hydrated cement paste and binding with the aggregates as evident in Field Emission Scanning Electron Microscopy (FESEM) micrographs of Fig. 5(a), Fig. 5(b) and Fig. 5(c) (Kardon, 1997; Ohama, 1995; Van Gemert et al., 2005). On the other hand, specimen incorporating 5% of Vinyl acetate effluents in concrete gave lesser compressive strength compared to 2.5% and this was due to the polymer volume contents that prohibited the cement grains to attach with water for cement hydration.

The compressive strength increased as curing time increases. For instance the 28 days compressive strength of the control mix had an increase of 45.3% over the 7 days. Similar results show increases of 28.99% and 44.16% for mix with 2.5% and 5% polymer Vinyl acetate effluents concrete. These indicate that the reaction of the cement continued over the days. It can also be seen that the mix with 2.5% polymer waste had higher early strength development.

#### 3.6 Temperature Rise and Heat of Hydration

Figure 6(a) showed the results of temperature rise and time of the control and polymer modified concrete Vinyl acetate effluents as stated in Table 6. It is observed that the trends of the temperature rise are almost similar. At the initial stage, the temperature rise of the entire test specimen where almost equal.

Table 5.	Workability	and	Compressive	Strength

Type of concrete	Slump (mm)	7-days Compressive Strength (Mpa)	28-days Compressive Strength (Mpa)	Compressive Strength Ratio (%)
PMCVA 0%	75	24.15	35.09	45.30
PMCVA 2.5%	90	31.87	41.11	28.99
PMCVA 5%	135	22.78	32.84	44.16

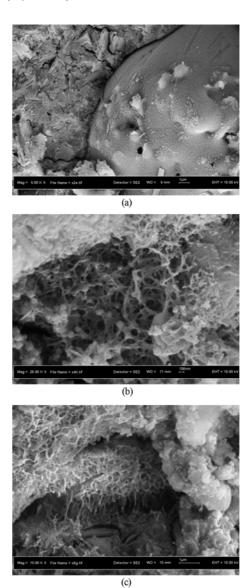


Fig. 5. (a) FESEM Micrographs of PMCVA 0% at 28 days, (b) FESEM Micrographs of Polymer Film of PMCVA 2.5% at 28 days, (c) FESEM Micrographs of Polymer Film of PMCVA 5% at 28 days

As the test duration increases the hydration effect among all the concrete mixes can be identified. Concrete incorporating Vinyl acetate effluents with 2.5% and 5% showed a reduce peaks of temperature rise by 1.8% and 4.9% compared to the control respectively. However, after reaching the peak temperature, all the temperature of the mixes drop gradually until it reached the stable condition. Similar results were observed from other studies showing the peak temperatures for control specimen are to be similar with recorded values in this study (Pofale *et al.*, 2013; Awal and Shehu, 2013; Atis, 2002).

The results of rate of heat of hydration of the specimens calculated from the recorded temperature rise and plotted as a function of time is shown in Fig. 6(b). The rate of heat of hydration shows similar trends to those of previous calorimetric studies. The peak rates of heat of hydration of specimens in the

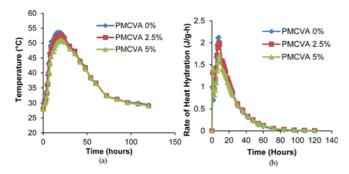


Fig. 6. (a) Heat of Hydration of Concrete with Vinyl Acetate Effluents, (b) Rate of Heat of Hydration as a Function of Time

Table 6. Temperature and Time of Concrete with Vinyl Acetate Effluents and Control Specimen

Materials/properties	PMC VA 0%	PMC VA 2.5%	PMC VA 5%
Initial Temperature (°C)	29.8	28.0	27.9
Peak Temperature (°C)	53.6	52.6 (1.8%)	51.0 (4.9%)
Time since mixing to peak temperature (h)	16	19	21

current study are 2.12 J/gh, 2.0 J/gh and 1.711 J/gh for 0%, 2.5% and 5% at 8 and 9 hours respectively. A previous study using cement using cement incorporating polymer Styrene acrylic ester shows rate of heat of hydration to be 2.70 J/gh and 1.70 J/gh at 8 and 7 hours for 0% and 5% specimens respectively (Wang and Shi, 2014). A study of addition of 3% Ethylene vinyl acetate to cement mortar (Liu *et al.*, 2013) also gave a comparable heat of hydration.

The reduction of the peak temperature is influenced by the incorporation of Vinyl acetate effluents possibly due to the presence of organic compounds and surfactants in the mixes. Hydration process occurs when typically cement particles react with the presence of water. The reaction is mostly exothermic and generates heat. However, by the addition of polymer in the mixes, the hydration of the cement is retarded. The retardation of the cement is possibly due to the interaction between polymers and ions (Ca<sup>2+</sup>, SO<sub>4</sub><sup>2-</sup> and OH<sup>-</sup>) released by the cement during hydration. Vinyl acetates comprise of carboxylic acid group which appears to interact with Ca<sup>2+</sup> in the pore solution and this reaction is known as saponification (Larbi and Bijen, 1990). The dispersant of polymer Vinyl acetate in alkaline medium releases acetate anion which react with C<sub>2</sub>S and C<sub>3</sub>S to form calcium acetate in the solution

The polymer Vinyl acetate may suffer hydrolysis when dispersed in alkaline medium that release acetate anion,  $CH_3COO^$ which react with  $Ca^{2+}$  ion from  $C_2S$  and  $C_3S$  to form an calcium acetate,  $(Ca(CH_3COO)_2)$  in the solution. The amount of Portlandite  $(Ca(OH)_2)$  reduced as a result of its consumption by the polymer, which appear to be more related to lowering cement hydration (Betioli *et al.*, 2009; Gomes and Ferreira, 2005; Silva *et al.*, 2002). On the other hand, incorporation of Vinyl acetate polymer forms a protective skin around the cement grains, which delayed the contact between cement particles and water in mixing. Consequently these extend the setting time of the mix and prolong the hydration of the concrete (Su *et al.*, 1991) thus, reducing the temperature thus in the mass concrete.

# 4. Conclusions

Based on the experimental conditions observed in the present study, the following conclusions can be drawn.

- 1. Vinyl acetate effluents functional group has a similar chemical structure with the pure Vinyl acetate latex which may give optimum condition regarding to the film formation in the mixes.
- 2. Setting time increases as the percentage of Vinyl acetate effluents increases as compared to the control.
- 3. The higher the percentages of Vinyl acetate effluents, the lower the heat of hydration of concrete compared to the control specimen. These may reduce potential crack propagation in the concrete.

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