Synthesis and Analysis of a Viscosity Modifying Admixture based on Acrylamide and 2-Acrylamide-2-methylpropanesulfonic acid

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Abstract—Viscosity modifying admixture (VMA) can be employed to avoid the segregation of concrete. Here, we present a type of viscosity modifying agent (B2), prepared by free radical polymerization between acrylamide (AM) and 2-acrylamide-2-methylpropane sulfonic acid (AMPS). The structure and composition were characterized by FT-IR, NMR and light scattering. Hydroxy-propyl-methyl cellulose (HPMC) was used as a comparison to determine the suitability of B2 as a VMA. The results show that B2 is superior to HPMC, and can improve not only the homogeneity and bleeding resistancy, but also increase the compressive strengths of the concrete by 4 to 5 MPa across all testing periods, up to 28 days. A highlight of this admixture is the effective regeneration of nonusable concrete to its non bleeding state with delayed addition. Finally, a mechanistic analysis of the viscosifying effect, which induced the increase in compressive strength is postulated in this article.

1. Introduction
The fresh concrete has to fulfill contradictory requirements of high flowability when it is being cast, and stability when it is at rest, in order to prevent segregation. High flowability is achieved using high performance of water reducer, while stability against segregation is achieved either by using a large quantity of fine materials or by using an appropriate viscosity agent (VMA) \cite{1,2}. In addition, self-compacting concrete (SCC) has higher requirements on the fluidity and stability of concrete than traditional concrete, so VMA becomes a necessary admixture for the SCC \cite{3}; VMA has been widely used to enhance the cohesion and stability of cement systems. The increase in VMA dosage is shown to exhibit a reduction in mini-slump and an increase in plate cohesion meter, Yield stress and plastic viscosity \cite{4}. VMAS can be divided into two categories according to their chemical composition, one is inorganic chemicals and the other is organic chemicals. Inorganic VAMs are as following: natural zeolite \cite{5}, silica fume \cite{6}; Organic VMAs have microbial polysaccharides, such as diutan gum, welan gum, cellulose derivatives (methyl cellulose, cellulose ether and cellulose filaments \cite{7}), bacterial cell walls (BCW) \cite{8} and rice husk ash (RHA) \cite{9} and so on. Inorganic VAM has the following advantages: (1) Inorganic VMA cause an acceleration of hydration and higher compressive strength at the age of 1 day \cite{10}; (2) Inorganic VMA causes the smallest change in the values of the air voids parameters. However, the dosage of inorganic VAM always exceeds 15% by weight of the cement, which leads to
low cost-effective performance; Organic VMA has the following advantages :(1) the dosage is much lower than the former, which is not more than 0.05% of the cement; (2) VMA was found to be very effective in reducing both crack area and crack width, and the crack reductions were correlated with surface tension, bleeding and weight changes [11]. The mechanism of action in each case is different and related to its molecular structure. The mechanism of action in each case is different. In the case of welan gum and cellulose, the long chain polymer molecules adhere to the periphery of water molecules, thus imbibing and fixing a part of the mixing water. Some other VMAs adsorb on cement particles and increase viscosity by promoting inter-particle attraction [12].

At present, organic VMA has been extensively and deeply studied by scholars. For a given dosage of VMA, diutan gum showed a high apparent viscosity than welan. For any given dosage of the super plasticizer, the diutan gum grouts a greater yield value and apparent and plastic viscosity than welan gum [13,14]. Cellulose ether adsorption seems to slow down the nucleation of calcium silicates at the surface of the cement grains in the tens of minutes following mixing, and bridge the cement grains by adsorbed ether molecule[15]. Cellulose ethers have the most high water retention capability. Increasing cellulose ethers addition will decrease slip-resistance[16]. According to the literature, the disadvantages of organic VMA also were found: (1) The organic VMA show almost no influence on early cement and the development of compressive strength [17]. The initial setting time is shown to be delayed by the incorporation of high range water reducer and VMA with the latter additive exhibiting a greater influence on retardation of setting [18] (2) organic VMA causes an happens in the content of the air pores;

2. Research Significance
As we knew that organic VAM has low dosage, good cost-efficiency, but which leads to reduce the concrete compressive strength[19]. In the article, a new organic VMA was studied based on the molecular design by introducing sulfonic group in order to avoid retarding effect on the concrete, as the sulfonic group was not chelated by calcium ions, and thus will not affect the compressive strength of the concrete; The introduction of amino group to the molecular structure would improve the hydrophilicity of molecules and early strength of concrete; The linear soft polymer was designed to block water molecules in mortar.

3. Experimental Investigation
3.1 Raw materials and instruments
Acrylamide (AM), industrial grade, purity> 99.0%; 2-acrylamide-2-methylpropyl sulfonic acid (AMPS), industrial grade, purity > 98.0%; Acetone, analytically pure; Azo disobutamidine hydrochloride initiator, industrial grade; Deionized water; Nitrogen, 99.9999% purity.

There were two types of chemical admixture used in the concrete experiment, including ether based polycarboxylate superplasticizer (GK-3000) and VMA. The GK-3000 behaves a water reduction of 30% and a solid content of 21%. Two VMAs chosen for this study are hydroxypropyl methylcellulose (HPMC) with a relative molecular mass of 1.5*10^6g/mol and a new type of VMA by self-made. Both the VMAs are water solution with mass fraction of 2%.

| Table 1 Chemical and physical properties of cement and coal ash | SiO₂(%) | Al₂O₃(%) | Fe₂O₃(%) | CaO(%) | SO₃(%) | MgO(%) | K₂O(%) | Na₂O(%) | LoI(%) | Specific surface area(cm²/g) |
|---------------------------------------------------------------|---------|---------|---------|--------|--------|--------|--------|---------|--------|-----------------------------|
| Cement                                                        | 22.12   | 4.75    | 3.14    | 64.3   | 2.12   | 1.91   | 1.02   | 0.24    | 1.6    | 3410                        |
| Fly ash                                                       | 54.1    | 27.4    | 6.35    | 2.18   | 0.14   | 0.78   | 1.61   | 0.35    | 92.91  | 3600                        |
3.2. **Synthesis of AM-AMPS copolymer**

At first, 120 mL of deionized water was placed in a 500 mL three-neck round bottom flask equipped with a stirrer, mercury thermometer. After the deionized water in the flask was heated to 60°C, purged N₂ into the water for 25min. Next, 15.6 g of AM and 5.3 g AMPS were added in the flask. Then, 0.03 g azo disobutamidine hydrochloride (initiator) was dissolved in the other 30 mL of deionized water, and was fed continuously into the reaction vessel over a period of 2 h at 60 °C using a peristaltic pump. When addition of the solution was completed, the mixture was curing for another 2 h to finalize the reaction. After that, another 200 mL deionized water and 3 g 30wt% NaOH solution were fed into the vessel, and the stirrer was accelerated in order to dissolve the production. At last, the solution was pure into 725 mL of deionized water, which was stirred for 20min, then a new VMA was obtained and named as B₂.

3.3 **Performance test method**

3.3.1 **FT-IR measurement**

Perkin Elmer Spectrum 100 Fourier transform infrared spectrometer is used to perform the test by using the thin film method with the resolution of 1cm⁻¹ and the scanning times of 32 times.

3.3.2 **NMR measurement**

The hydrogen spectra of the refined samples were measured by INOVA 400 NMR apparatus with D₂O as the solvent and methyl silane (TMS) as internal standard.

3.3.3 **Light scattering measurement**

Static and dynamic light scattering were measured by Brookhaven light scatterer. The wavelength of the laser used for light scattering is 532nm, and the angle of the static light scattering is from 15° to 155°. The scattering Angle selected for dynamic light scattering is 90°.

3.3.4 **Rheological measurement**

HAAKE RS150 torque rheometer was used for steady-state rate scanning and dynamic frequency scanning experiments. All tests were carried out at 25°C. The diameter and cone Angle of the lamina were 40mm and 4°, respectively, and the plate spacing was 150 μm.

3.3.5 **Concrete test**

In order to compare the effect of B2 and HPMC on properties of concrete, concrete have been mixed with a water-cement of 0.46, 0.29kg PCE (made by Shijiazhuang Chang’an Yucai Building Material Co., LTD, China) is added to 1m³ concrete to make the concrete slightly bleeding, and the slump flow is 600mm. The dosage of B2 is 0.01%, 0.015%, 0.03%, 0.05% of concrete, and the dosage of

| Table 2 Grading of artificial sand |
|-----------------------------------|
| Sieve diameter (mm) | sieve passing (g) | sieve percentage passing (%) | cumulative percentage passing(%) | fineness modulus |
| 4.75 | 6.50 | 1.3 | 1.3 |
| 2.36 | 98.00 | 19.6 | 20.9 |
| 1.18 | 93.50 | 18.7 | 39.6 |
| 0.600 | 89.00 | 17.8 | 57.4 | 2.70 |
| 0.300 | 71.00 | 14.2 | 71.6 |
| 0.150 | 52.00 | 10.4 | 82.0 |
| 0.075 | 48.50 | 9.7 | 91.7 |
| Sieve bottom | 41.50 | 8.3 | 100.0 |
HPMC(2wt%) solution is 0.01%, 0.03%, 0.05%, 0.07% of concrete. The mixture proportions are given in Table 3. Cementitious materials and some of the water were firstly added in mixer and stirred for 30 s. Water containing dissolved VMA and SP was then poured and mixed for 60 s. Finally, gravels were added and mixed for another 60 s. The prepared fresh concrete mixture was measured by the slump flow test, bleeding test, flow time of mixture from the inverted slump cone, air content test, in accordance with Chinese Standard GB/T 50080-2016. Furthermore, compressive strength on 100×100×100 square (at 3, 7, 28 days) were assessed. In order to study the effect of B2 on regeneration of nonuseable concrete to its non-bleeding state with delayed addition. Cementitious materials and some of the water were firstly added in mixer and stirred for 30 s. Water containing SP which dosage is 1.5% of concrete to make the concrete bleeding was then poured and mixed for 60 s. Then, gravels were added and mixed for another 60 s. Finally, B2 which dosage is 0.2% of concrete was add and mixed for 30s. The prepared fresh concrete mixture are also measured by the slump flow test, bleeding test, flow time of mixture from the inverted slump cone, air content test.

| Table 3. Mixture proportion of concrete |
|----------------------------------------|
| W/C | Unit weight (kg/m³) |
|     | C  | F  | S  | G  | W  |
| 0.46 | 290 | 70 | 805 | 1025 | 165 |

4. Experimental Results and Discussion

4.1 Composition and structure of copolymer

4.1.1 Infrared and NMR spectra

As shown in Fig.1, there is a strong peak at 3432.18 cm⁻¹, which is the stretching vibration absorption peak of N-H on the amide group. The stretching vibration absorption peak of methylene at 2931.33 cm⁻¹ is a moderate intensity peak. The bending vibration absorption peaks of -CH₃ at 1414.74 cm⁻¹ and 1453.96 cm⁻¹ are two weak peaks, and the stretching vibration absorption peak of carbonyl group at 1639.19 cm⁻¹ is strong. Because the sample had been extracted with acetone as the solvent for 72 hours before the infrared test, the monomer and low molecular homopolymers had been removed, so the infrared results preliminarily indicated that AM and AMPS had formed a copolymer.
In Fig.2, a wide peak appears at 1.47ppm of chemical shift, which should be the result of the superposition of two -CH₃ (c) peaks in AMPS unit. Due to the influence of electron-withdrawing group SO³⁻, the chemical shift of -CH₂ increases. The chemical shifts of -CH₃ were found at 2.2ppm and 2.5ppm, and the peak strength at 2.2ppm was the largest (except the solvent peak), as could be attributed to the methylene peak (a) of the acrylamide unit contained the most hydrogen. The peak at 2.5ppm was assigned to the methylene peak (d) in the AMPS unit, whose chemical shift was greater than that of the -CH peak (e) in the AMPS unit, which was also affected by the anion of sulfonic acid group. However, the chemical shift of methylene peak (b) at 2.7ppm, which was attributed to acrylamide. Based on the above analysis, it is further proved that the AMPS-AM copolymer has been successfully synthesized.

4.2 Light scattering characterization
The zimm diagram of the copolymer in the pore fluid of the cement slurry determined by static light scattering is shown in Fig.3. The average molecular weight (Mₕ), the mean square root mean square radius of rotation (Rg) and the second dimension coefficient (A₂) of the B₂ sample were obtained, which are shown in Table 4.
The testing results of static light scattering (Figure 3 and Table 4) show that the Mw of the B2 reached 1.73 x 10^6 g/mol, whose root mean square radius of gyration is 61.1 nm. The second virial coefficient between B2 and the pore fluid is 3.3 x 10^{-3} cm^3 mol/g^2. The above results indicate that the copolymer contained in B2 has a high molecular weight and a strong interaction with water molecules in the pore solution, and the linear molecules are speculated that the conformation of the polymer is relax in the solution.

Further dynamic light scattering test was performed on the pore solution of the copolymer, and the concentration of the polymer solution was 150mg/L. The test results were shown in Fig.4. It can be seen from the figure that the distribution of copolymer solution particle size (D) is multi-dispersive, which is distributed in the range of 2.0-2.8nm, 26.3-36.2nm and 94.5-210.2nm, respectively, and the number of particles in the range of 94.5-210.2nm is the largest. The hydraulics radius of the polymer in solution, Rh, is equal to D/2. Rh can be considered as the radius of the equivalent hard sphere with the same translational diffusion coefficient as the polymer. If the polymer chain is an extended Gaussian chain, the solvent molecules in the space occupied by the polymer chain do not move with the polymer chain when the polymer chain is diffused. At this point, Rh is much less than Rg, indicating that the polymer molecule is rigid. But when the polymer chain forms a curled ball, the solvent molecules move along with the polymer. At this condition, Rh is greater than Rg. Therefore, Rg/Rh of scattered particles is used to investigate the microscopic conformation of scattered particles in solution [7]. The average hydraulics radius Rh is 61.1nm (123.1/2), and the ratio of Rg/Rh is 1.0. The difference between them is not significant, which indicates that the copolymer presents a flexible curl shape and can move together with the solvent molecules.
Based on the above analysis, it is concluded that amide units introduced in the copolymer molecular chain greatly enhances interaction between water molecules and the polymer, which leads to more water molecules controlled by the polymer; Rigidity and ion repulsion of sulfonic acid in the molecular structure in the pore fluid can increase of Rh and are beneficial to improve the water retention performance.

4.3 Rheological properties

The steady-state rheological diagram of the copolymer in the pore fluid was shown in Fig.5, and the concentration of the pore solution is 1500mg/L. It can be seen from the figure that the viscosity of the synthesized copolymer in the pore solution can reach more than 3 Pa.s at the low shear rate, while the viscosity of the HPMC in pore solution is above 4 Pa.s at the low shear rate, which indicates that both of them have good viscosity retention rate, namely good water retention when they are static. On the rheological curve of shearing and viscosity, the viscosity of copolymer and HPMC in the pore fluid increased slowly with the increase of shear rate at beginning, which showed a tendency of shear-thickening. Then a plateau appeared and began to decrease with the increase of shear rate. At high shear rate, the apparent viscosity of the copolymer in pore solution decreases linearly with the increase of shear rate, which the characteristic property of shear-thinning was appeared. The viscosity of HPMC decreases less than that of copolymer at high shear rate. The property of the shear-thickening of the two VMAs at low shear rates can be explained by the orientation and entanglement effects of
the molecular chains. In the pore solution, the molecular chains of the VMA are entangled or hydrogen-bonded to form a series of physical crosslinking points. Due to the thermal movement of the molecules, these physical crosslinking points are constantly disintegrated and formed, and their positions are constantly changing. In the low shear rate region, the physical crosslinking points destroyed by shearing can be reconstructed in time, and the molecular orientation increases the viscosity of the copolymer solution. When the shear rate increases to a certain extent, the destructed speed of the physical crosslinking points is greater than its reconstructed speed, so that the friction resistance decreases and the solution appears shear-thinning behavior. In addition, due to the stronger intermolecular interaction of HPMC, the physical cross-linking network formed in solution has stronger shear resistance than linear copolymer molecules.

4.4 Properties of concrete

In order to compare the effect of B2 and HPMC on properties of concrete, concrete have been mixed with a water-cement of 0.46, 0.29kg PCE (made by Shijiazhuang Chang’an Yucai Building Material Co., LTD, China) is added to 1m³ concrete to make the concrete slightly bleeding, and the slump flow is 600mm.

![Fig.6 Curves of bleeding rate versus dosage for concrete added with B2 and HPMC](image)

Fig.6 gives the Curves of bleeding rate versus dosage for concrete added with B2 and HPMC. It can be seen that the bleeding rate of concrete decreased significantly after treated with B2, and decrease to 0% when the dosage of B2 is 0.015%. However, the bleeding rate of concrete is not changed when the dosage of HPMC is low, and it dropped to 0 while the dosage of HPMC is 0.05%, which is higher than B2. It can be explained, that hydrophilic groups of VMA molecules (such as the hydroxypropyl in HPMC, the acylamino in B2) can adsorb and associate water molecules via hydrogen bond. Then Van der Waals attractive forces produced by adjacent B2 chains can retard the free motion of water therein and even block water molecules as the chains are intertwined, thus presenting a network at low concentration. Whereas HPMC molecules had shorter and semi-rigid main chain composed of glucosyl rings, the chains were difficult to intertwine. Therefore, the free motion of water is retarded only when the dosage of HPMC is high, the bleeding rate of concrete is also decreased.
Fig. 7 Curves of flow time versus dosage for concrete added with B2 and HPMC

The curves of flow time versus dosage for concrete added with B2 and HPMC are presented in Fig. 7. It can be seen that the flow time of concrete is decreased and then increased with the increase of B2 dosage, and the flow time attain the minimum when the dosage of B2 is 0.03%. But the flow time of concrete ascend with the increase of HPMC dosage. It is all known that the flow time of concrete has a great relationship with the plastic viscosity and workability of concrete. As 2.2.1 said, B2 chains are entangled or hydrogen-bonded to form a series of physical crosslinking points when concrete does not flow, consequently the workability of concrete is excellent. When concrete flows, mortar added with B2 appears shear thinning behavior, thus the increase of plastic viscosity is not obvious. Therefore, with the increase of B2 dosage, the flow time does not increase. Whereas HPMC molecules had shorter and semi-rigid main chain, and the free motion of water is retarded only when the dosage of HPMC is high, otherwise, mortar added with HPMC does not appear shear thinning behavior significantly, thus the plastic viscosity of concrete increases significantly as HPMC dosage is high. So that the flow time of concrete increase with the increase of HPMC dosage.

Fig. 8. Curves of slump and slump flow versus dosage for concrete added with B2 and HPMC
As it can be seen in Fig.8, with the increase of B2 dosage, the slump and slump flow of concrete do not change much, but with the increase of HPMC, the slump and slump flow of concrete decreased. Due to the molecular structure, there is little effect of B2 on the slump and slump flow of concrete. However, on one hand, the plastic viscosity of concrete increases significantly as HPMC dosage is high, on the other hand, due to the introduction of hydrophilic group (hydroxypropyl) and hydrophobic group (methoxy) in its molecular structure, when its dosage is high, it will introduce bubbles into concrete, further increasing the viscosity of concrete. Therefore, the concrete slump and slump flow decrease with the increase of HPMC content.

Fig.9. Curve of concrete air content versus dosage for concrete added with B2 and HPMC

Fig.9 shows the variation curve of concrete air content with B2 and HPMC content. With the increase of B2 content, the air content of concrete has no obvious change, but with the increase of HPMC content, the air content of concrete keeps increasing. This also proves that the introduction of hydrophilic (hydroxypropyl) and hydrophobic (methoxy) groups into the molecular structure of HPMC will introduce bubbles into the concrete. The increase of concrete air content will inevitably lead to the decrease of concrete strength.
As can be seen from Figure 10, B2 can increase the strength of concrete, especially the 3d strength. HPMC will reduce the strength of concrete, and with the increase of HPMC content, concrete 3d, 7d and 28d strength continues to decrease, this is mainly because with the increase of HPMC content, concrete air content continues to increase, resulting in a continuous decline in strength. The air content of the concrete mixed with B2 does not change significantly, and B2 contains amino, the N atom in the amino has a pair of unshared electrons, it is easy to form covalent bonds with metal ions, complex, and to form a relatively stable complex with metal ions. These complexes form a number of soluble zones in the solution, thus increase the diffusion rate of the hydration products and improve the early strength of concrete.

![Fig. 10. Curve of concrete strength versus dosage for concrete added with B2 and HPMC](image)

(a: 3d, b: 7d, c: 28d)

Fig. 10. Curve of concrete strength versus dosage for concrete added with B2 and HPMC

![Fig. 11. State of bleeding concrete before and after addition of B2](image)

(A: before addition, B: after addition)

Fig. 11. State of bleeding concrete before and after addition of B2
Table 5 The performance of fresh concrete

| Sample | Bleeding rate (%) | Flow time (s) | Slump (mm) | Slump flow (mm) |
|--------|-------------------|---------------|------------|-----------------|
| Before | 1.6              | 11.2          | 240        | 620             |
| After  | 0                | 6.7           | 240        | 545             |

As can be seen from Fig. 11 and the table 5, after the addition of B2 into the bleeding concrete, the concrete aggregate and slurry are no longer separated, the flow time of concrete reduces, and the concrete shows good workability.

5. Conclusion
A new type of viscosity modifying agent (B2) was prepared by free radical polymerization between acrylamide (AM) and 2-acrylamide-2-methylpropane sulfonic acid (AMPS) in the article. The structure and composition were characterized by FT-IR, NMR and light scattering. Hydroxy-propyl-methyl cellulose (HPMC) was used as a comparison to determine the suitability of B2 as a VMA. The results show that B2 is superior to HPMC, and can improve not only the homogeneity and bleeding resistancy, but also increase the compressive strengths of the concrete by 4 to 5 MPa across all testing periods, up to 28 days. A highlight of this admixture is the effective regeneration of nonuseable concrete to its non bleeding state with delayed addition. Finally, a mechanistic analysis of the viscosifying effect, which induced the increase in compressive strength is postulated in this article.

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