Retention behavior of complexes formed by carboxymethyl cellulose and a cationic polymer in pulp sheets for wet wiper

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KEYWORDS: CMC, retention, PTMMAC, Wet wiping sheet, Polyion complex, Zeta-potential.

SUMMARY: In the case of wet wiper sheets, relatively large amounts of carboxymethyl cellulose (CMC) have to be retained in pulp sheets to have enough wet tensile strength in use and high water-disintegrability after use. For this purpose, CMC-containing pulp sheets were prepared by a dual polymer system using poly[*N,N,N*-trimethyl-*N*-(2-methacryloxyethyl)ammonium chloride] (PTMMAC). Retention behavior of PTMMAC and CMC in pulp sheets was studied in terms of addition level and molecular weight of PTMMAC used as the cationic retention aid of CMC. When 5% PTMMAC and 5% CMC (both percentages are based on dry weight of pulp) were added in this order to pulp slurries, ~80% and ~90% of PTMMAC and CMC added, respectively, were retained in the pulp sheets, and these PTMMAC/CMC/pulp sheets gave the highest dry tensile strength in the sheets prepared with 1-7% PTMMAC regardless of molecular weight of PTMMACs. The retention behavior of PTMMAC and CMC were well explainable by surface charges of pulp fibers and colloidal particles in the pulp slurries or their charge balances. The molecular weight of PTMMAC had almost no influence on wet strength of the PTMMAC/CMC/pulp sheets, but significantly affected their water-disintegrability behavior; the lower the molecular weight of PTMMAC, the higher the water-disintegrability of the sheets.

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Wet wiper sheets for cleaning toilet or bathroom have been commercially available as household and disposable commodity goods. When the wet wiper sheets are made from bleached softwood kraft pulp by papermaking process, sufficient wet tensile strength in use and water-disintegrability after use should be provided to the sheets by wet-end additives and impregnation solutions to the sheets. Because the wet strength and water-disintegrability are somewhat opposite nature for pulp sheets, various patents to manufacture wet wiping sheets disposable in toilet water have been published (e.g. Kakiuchi et al. 1993, 1994). Empirically, it has been known that carboxymethyl cellulose (CMC)-rich pulp sheets have potential to increase in wet strength to a certain level using ethanol and calcium chloride as the components in liquid impregnates containing water-based detergents.

Several procedures to efficiently attach anionic CMC molecules to anionic pulp fibers have been reported so far. CMC molecules with low degrees of substitution (DS) irreversibly adsorb on pulp fibers by particular treatments (Laine et al. 2000; Blomstedt et al. 2007; Rakkolainen et al. 2009) or under specific papermaking conditions (Gondo et al. 2006). However, in these cases, the adsorbed amounts of CMC are less than 1.5% on dry weight of pulp. So-called dual polymer systems using cationic polymer and CMC are alternatives to retain CMC molecules in pulp sheets, which are primarily carried out to improve dry strength of the sheets (Hubbe et al. 2003; Feng, Pelton 2007; Hubbe et al. 2009; Fatehi et al. 2010). However, the addition levels of CMC were less than 2% on dry weight of pulp also in these cases. Dual polymer systems have been studied for enhancing efficient and homogeneous retentions of functional fillers such as iron powder and TiO_2 catalyst in paper-like sheets (Ichiura et al. 2001; Fukahori et al. 2006; Koga et al. 2008; Kumamoto et al. 2009).

A previous report describes CMC-rich pulp sheets prepared by paper-making technique using a dual polymer system with poly[*N,N,N*-trimethyl-*N*-(2-methacryloxyethyl) ammonium chloride] (PTMMAC) as cationic polymer (Uematsu et al. 2011). PTMMAC/CMC/pulp sheets were prepared with 5% PTMMAC and 5% CMC, and chemical structures of these additives in the sheets were analyzed. When the PTMMAC/CMC/pulp sheets were soaked in solutions consisting of ethanol, water and calcium chloride ($EtOH/H_2O/CaCl_2$) with a weight ratio of 75:24:1, almost all PTMMAC and CMC molecules in the sheets turned to PTMMAC-N^+Cl^- and CMC-COOCaCl structures without dissolution into the soaking solution. Thus, PTMMAC, CMC and calcium contents in the sheets could be determined on the basis of these PTMMAC and CMC structures from analytical data such as nitrogen, calcium and chlorine contents.

In this paper, retention behavior of PTMMAC and CMC in pulp sheets is studied in detail in terms of PTMMAC addition level, molecular weight of PTMMAC and surface charges of pulp fibers and colloidal particles in PTMMAC/CMC/pulp slurries using some analytical methods. Moreover, the effect of molecular weight of PTMMAC on dry and wet strengths and water-disintegrability of the PTMMAC/CMC/pulp sheets are investigated.

Materials and methods

Materials

A commercial mixed softwood bleached kraft pulp with 720 ml Canadian Standard Freeness was used without beating in handsheet-making after removal of fines fraction using a 150-mesh wire screen. A commercial and salt-free CMC with a DS of 0.963 and weight-average degree of polymerization (DP_w) of 640 was kindly provided by Daiichi-Kogyo Seiyaku Co., Ltd. PTMMAC samples with different DP_w values were prepared from TMMAC and 2,2-azobis-2-amidinopropane (initiator, V-50) basically according to the previously reported method (Uematsu et al. 2011) but with different TMMAC/initiator weight ratios, and used after dissolution in water and freeze-drying. One % (w/v) solutions of PTMMAC and CMC were prepared by dissolution in distilled water, and used as additives in sheet-making. Standard solutions of poly(diallyl-dimethyl ammonium chloride) and potassium poly(vinylsulfate) (Wako Pure Chemicals Co., Japan) with 2.5 meq. l^{-1} were used in polyelectrolyte titration after dilution with distilled water.

Sheet-making

Designed amounts of PTMMAC and CMC solutions were added in this order to a 1% pulp slurry under continuous stirring at 300 rpm. The lapse of time between the PTMMAC and CMC additions was set to be 1 min. In a separate case, PTMMAC and CMC were first added in this order to water followed by addition of the wet pulp. Handsheets with a basis weight of 60 g m^{-2} were prepared from the pulp slurry with tap water at pH 7.3 and 0.25 mS cm^{-1}, according to TAPPI T 205 sp-95 (2005). The wet-pressed webs were dried at 100°C for 2 min using a rotary drum-dryer. The handsheets thus prepared were conditioned at 23°C and 50% relative humidity for more than one day.

General analyses

Water-disintegrability of the sheets was evaluated according to the Japanese Industrial Standard (JIS) P4501 (1993). Water (300 ml) in a beaker was first stirred at 600 rpm with a disk-type magnetic stirrer bar 35 and 12 mm in diameter and thickness, respectively. When a square-cut sheet with 60×60 mm was dropped in the stirred water, the stirring speed initially decreased below 500 rpm and then increased as the disintegration of the sheet proceeded in water. The time required for increasing the stirring speed to 540 rpm was measured 5 times for each sample, and the average value was defined as "time required for dispersion of the sheet to fibers in water". Details of the following analyses were described in the previous paper (Uematsu et al. 2011): ionic charge densities of CMC and PTMMAC, DP_w values of PTMMACs, dry tensile strength of handsheets, wet tensile strength of handsheets after soaking in mixtures of $EtOH/H_2O/CaCl_2$ with various weight ratios, and PTMMAC-N^+ and CMC-COO^- contents in the sheets.

Characterization of pulp fibers and colloidal particles in PTMMAC/CMC/pulp slurries

The pulp (1.2 g) was stirred in 0.002 M $NaNO_3$ (120 ml), which had the same pH and conductivity values as those of the tap water used in handsheet-making. 1-7% PTMMAC and 5% CMC (both percentages were based on dry weight of the pulp) were added in this order to the pulp slurry. After being stirred for 3 min, the pulp slurry was filtered in a glass funnel (P-40 with pore size of 16-40 μm) by suction. The filtrate was diluted to 200 ml with 0.002 M $NaNO_3$, and then subjected to measurements of UV-vis light transmittance and zeta-potential/z-average particle size at 25°C using a Shimadzu UV-1700 and a Malvern Zetasizer 3000, respectively. Further, the pulp fiber mat formed on the glass funnel was washed thoroughly with 0.002 M $NaNO_3$ by filtration, and then the pulp mat was dispersed to fibers in 0.002 M $NaNO_3$ (500 ml). Zeta potentials of the pulp fibers were measured at 25°C by streaming potential method using a System Zeta Potential SZP04 (BTG Mütek GmbH, Germany) (Horvath, Lindström 2006).

Results and discussion

Retention of PTMMAC in sheet

Fig 1 shows the effect of PTMMAC addition level to pulp slurries on the PTMMAC-N^+ content in the sheets and the corresponding retention ratio. The lapse of time between the PTMMAC addition and sheet-making was set to be 1 min. The PTMMAC with DP_w of either 342 or 6450 was used as a sole additive to pulp slurries. Similar retention patterns were obtained for the two cationic polymers. However, the maximum and plateau PTMMAC-N^+ contents in the sheets at 1-5% addition levels were different to some extent; approximately 1.7 and 1.4 mg g^{-1} for the sheets prepared with the polymers having DP_w of 342 and 6450, respectively. It is well known that higher amounts of cationic polymers with lower molecular weights adsorb on negatively charged pulp fibers in water, because such polymers can penetrate also into small pores present on pulp

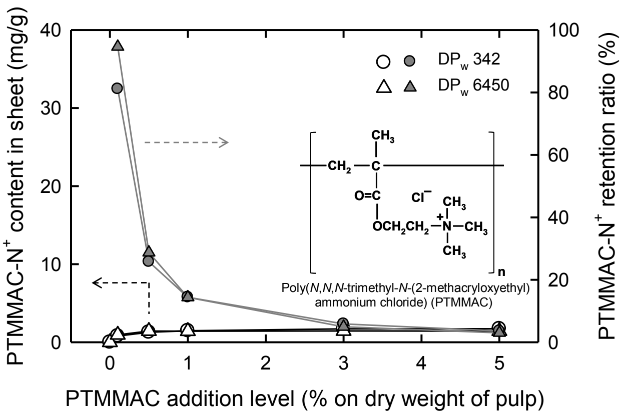

Fig 1. PTMMAC-N⁺ content and the corresponding PTMMAC-N⁺ retention ratio in sheets prepared with 0-5% PTMMAC.

fiber surfaces to be fixed there by electrostatic interactions (Lindström, Söremark 1976; Wågberg et al. 1988; Tanaka et al. 1990; Li et al. 2004).

However, the retention ratios were lower than 15% at 1-5% addition levels for both PTMMACs, showing that more than 85% of the added PTMMAC-N$^+$ electrolytes were present in the drainage water without adsorption on pulp fibers during the handsheet-making process. Thus, there were explicit limitations of the amount of PTMMAC-N$^+$ retained in the sheets, when only PTMMAC is added to pulp slurries.

Retention behavior of PTMMAC and CMC in sheets prepared by dual polymer system

The amounts of PTMMAC-N$^+$ retained in the sheets significantly increased by the dual system using CMC as the co-additive, regardless of the DP$_w$ values of PTMMACs (*Fig 2*). When 5% PTMMAC and 5% CMC were added in this order to pulp slurries, the PTMMAC-N$^+$ contents in the sheets reached approximately 30 mg g^{-1} for both PTMMACs with different DP$_w$ values, which corresponded to ~80% retention ratios. In contrast, when the addition levels of PTMMACs were 1, 3 and 7%, the PTMMAC-N$^+$ contents or the retention ratios were lower than those obtained for the sheets prepared with 5% PTMMAC and 5% CMC. The low PTMMAC-N$^+$ retentions were caused by imbalanced charges between cationic and anionic groups in the pulp slurries, as described later.

The CMC-COO$^-$ content in the sheets or the corresponding retention ratio versus the PTMMAC addition level are depicted in *Fig 3*. The patterns were similar to those for the PTMMAC-N$^+$ contents in *Fig 2*; the highest CMC-COO$^-$ contents/retentions were achieved at the 5% PTMMAC addition level, in which the retention ratios of CMC-COO$^-$ reached ~90%. The difference in DP$_w$ of PTMMACs had almost no influence on the CMC-COO$^-$ retention. Thus, the pulp sheets containing considerable amounts of CMC-COO$^-$ electrolytes, i.e. 35~38 mg g^{-1}, can be prepared by the dual polymer system using cationic PTMMAC as the co-additive.

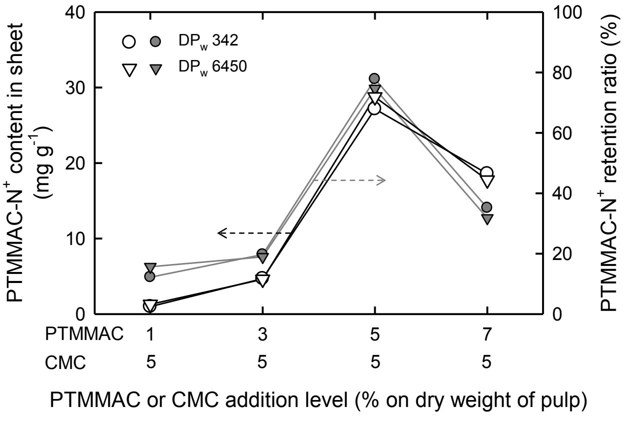

Fig 2. PTMMAC-N⁺ content and the corresponding PTMMAC-N⁺ retention ratio in sheets prepared by dual polymer system with 1-7% PTMMAC and 5% CMC.

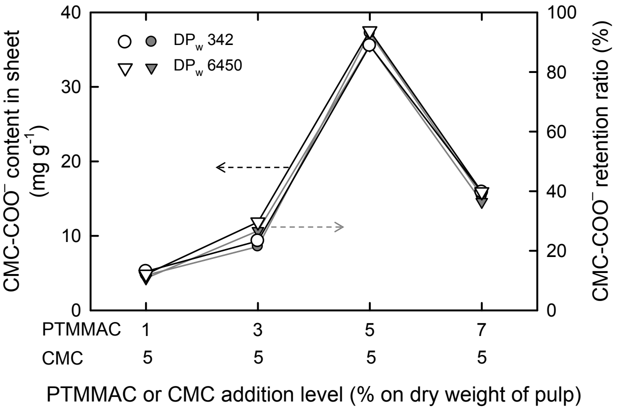

Fig 3. CMC-COO⁻ content and the corresponding CMC-COO⁻ retention ratio in sheets prepared by dual polymer system with 1-7% PTMMAC and 5% CMC.

The maximum amounts of PTMMAC-N$^+$ retained in the sheets were only 1.4-1.7 mg g^{-1}, when only PTMMAC was added to the pulp slurries (*Fig 1*). Hence, the high PTMMAC-N$^+$ and CMC-COO$^-$ contents in the sheets were supposed to be achieved by forming polyion complexes between the cationic and anionic polymers in the pulp slurries. In these cases, the charge balance between cationic PTMMAC-N$^+$, anionic CMC-COO$^-$ and anionic pulp fibers must be the primary factor influencing the surface charges of pulp fibers and polyion complexes formed *in situ*, which might result in the different retentions of the additives in *Figs 2* and *3*.

Fig 4 shows zeta-potentials of pulp fibers and colloidal particles in the pulp slurries, which were prepared with 1-7% PTMMAC and 5% CMC. As expected, both pulp fibers and colloidal particles had zeta-potentials close to zero, when 5% PTMMAC and 5% CMC were added, which consequently brought about the highest retentions of

PTMMAC-N$^+$ and CMC-COO$^-$ electrolytes (Hubbe et al. 2003).

Both pulp fibers and colloidal particles had positive surface charges at the 6-7% PTMMAC addition level. Electrostatic repulsion might act between pulp fibers and colloidal particles or between colloidal particles themselves, resulting in lower retentions of both PTMMAC-N$^+$ and CMC-COO$^-$ electrolytes. Contrastively, both pulp fibers and colloidal particles had negative surface charges at the 1-4.3% PTMMAC addition levels, resulting also in lower retentions of both electrolytes.

UV-vis light transmittances of the filtrates and the corresponding optical photos are presented in *Fig 5*. Expectedly, the light transmittance of the filtrate was the highest for the pulp slurry prepared with 5% PTMMAC and 5% CMC, because almost all PTMMAC and CMC electrolytes adsorbed on pulp fibers, forming polyion complexes. In contrast, the colloidal particles of PTMMAC/CMC polyion complexes were stably present in the filtrates by electrostatic repulsions at other PTMMAC addition levels. The PTMMAC/CMC colloidal particles formed in the pulp slurry were quite small in quantity at the 1% PTMMAC addition level, resulting in such a high UV-vis light transmittance in *Fig 5*. Thus, the retention behavior of PTMMAC-N$^+$ and CMC-COO$^-$ electrolytes observed in *Figs 2* and *3* is interpretable by charge balance between anionic and cationic groups present in the pulp slurries (Hubbe et al. 2003). In conclusion, such large amounts of anionic CMC-COO$^-$ electrolytes can be retained in pulp sheets by the dual polymer system used in this study.

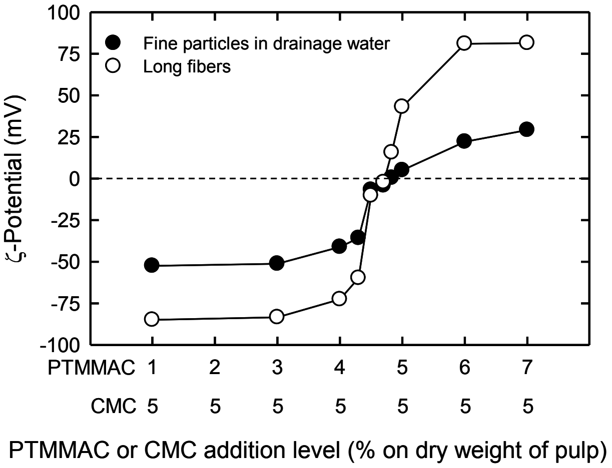

Fig 4. ζ-Potentials of pulp fibers and colloidal particles in pulp slurries. 1-7% PTMMAC with DP$_w$ 342 and 5% CMC were added to pulp slurries.

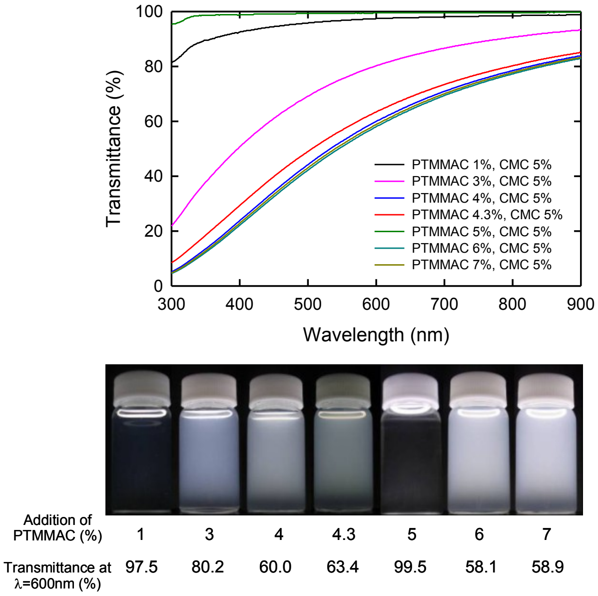

Fig 5. UV-vis light transmittances and the corresponding photos of filtrates of pulp slurries. 1-7% PTMMAC with DP$_w$ 342 and 5% CMC were added to pulp slurries.

Effects of addition level and DP$_w$ of PTMMAC on dry strength of the sheets

One of the properties required for wet wiper sheets made of pulp fibers is to have sufficient wet tensile strengths in use. Dry tensile strengths of more than 40 Nm g^{-1} are also needed for the sheets in manufacturing and embossing processes without web-break. Moreover, there is a clear link between wet and dry strengths of PTMMAC/CMC/pulp sheets (Uematsu et al. 2011); the higher the dry strength of the sheets, the higher the wet strength as well. Thus, dry tensile strength of the PTMMAC/CMC/pulp sheets provides an indication of the wet strength properties required for wet wiper sheets. Dry tensile strengths of the sheets prepared with 1-7% PTMMAC and 5% CMC are depicted in *Fig 6*. The highest dry-tensile strengths were expectedly observed for the sheets prepared with 5% PTMMAC and 5% CMC. The levels of such dry tensile strength were comparable to those prepared from beaten pulp of 400-500 ml Canadian Standard Freeness without any additives. Hence, the PTMMAC and CMC electrolytes retained as polyion complexes in the sheets play a role in improving inter-fiber bonds. Because the handsheets prepared with the PTMMAC with DP$_w$ of 6450 had slightly higher dry tensile strengths at 1, 3 and 5% PTMMAC addition levels than those prepared with PTMMAC with DP$_w$ of 342, PTMMAC molecules with higher DP$_w$ may have some contribution to higher dry tensile strengths of the sheets.

When the pulp slurry was prepared by the different addition sequence, in which PTMMAC and CMC were first added to water followed by the addition of pulp, dry tensile strength of the sheets (Sheet B in *Fig 6*) thus prepared was remarkably low particularly at the 5% PTMMAC and 5% CMC addition levels. The supernatant of the pulp slurry prepared by the normal PTMMAC/CMC addition

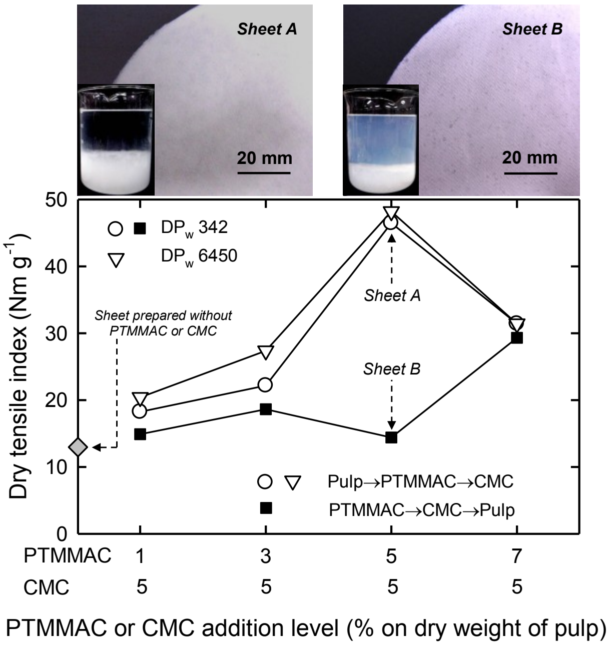

Fig 6. Dry tensile strength of sheets prepared with 1-7% PTMMAC and 5% CMC. The effects of addition order of additives on dry tensile strength and appearance of the pulp slurries and dried sheets are presented also in this figure.

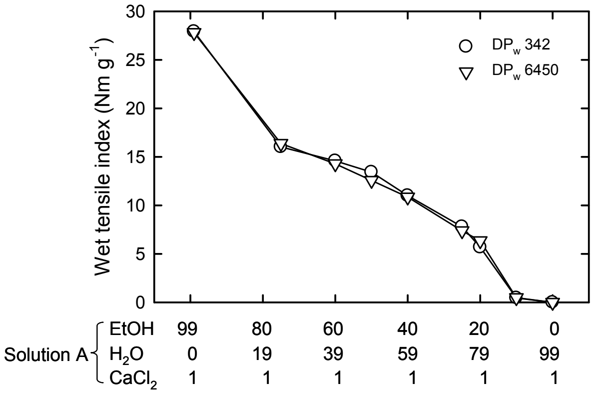

Fig 7. Wet tensile strength of sheets prepared with 5% PTMMAC and 5% CMC, after soaking in Solution A. PTMMACs with two different DPw values were used in sheet-making (Uematsu et al. 2011).

sequence was transparent, and the corresponding sheet surface (Sheet A in *Fig 6*) was smooth and had no visible particles. On the contrary, the supernatant of the pulp slurry prepared by the PTMMAC→CMC→ pulp sequence was turbid, and many and large particles due to PTMMAC/CMC polyion complexes easily visible to the naked eye were present on the sheet surface. Thus, in the normal addition sequence, i.e. pulp→PTMMAC→ CMC, the PTMMAC/CMC polyion complexes with smaller particle sizes are likely to form in the pulp slurries, and mostly and homogeneously adsorb on pulp fiber surfaces during the sheet-making process, resulting in high dry tensile strengths. Contrastively, when the PTMMAC and CMC were first added to water, large particles of polyion complexes were first formed in water, and these particles were then trapped on the sheets mostly by filtration effect during the sheet-making process, resulting in low tensile strengths.

Wet strength and water-disintegrability of the sheets

It was found that wet strengths of the PTMMAC/ CMC/pulp sheets were improved by soaking the sheets in EtOH/H_2O/$CaCl_2$ solutions (Solutions A) with suitable weight ratios of the three components. The influence of DP$_w$ of PTMMACs on wet strength of the sheets soaked in Solutions A are displayed in *Fig 7*. No clear difference in wet strength patterns were observed between the two PTMMACs; the wet strength of the sheets decreased with increasing the weight ratio of water in Solutions A at the soaking treatment regardless of the DP$_w$ values of PTMMACs. When 20-40% EtOH and 1% $CaCl_2$ were present in the soaking solutions, wet strengths of the sheets became more than 5 Nm g^{-1}, the level of which is sufficient for wet wiper sheets. Such Solutions A containing water-based detergents may be used as impregnation solutions into the PTMMAC/CMC/pulp sheets for wet wipers. The detailed mechanism to improve wet strength of the sheets by soaking in such Solutions A containing EtOH and $CaCl_2$ has been reported previously (Uematsu et al. 2011).

The CMC-COO$^-$ electrolytes present in the sheets forming polyion complexes with PTMMAC-N$^+$ electrolytes were found to be mostly turned to CMC-COOCaCl type-salt structures by exchanging the PTMMAC-N$^+$ electrolytes for CaCl$^+$ ions during the soaking treatment in Solutions A containing EtOH and $CaCl_2$. Degrees of dissociation of such CMC salts are restricted to some extent in the soaking solutions containing EtOH, resulting in wet strength development. When the sheets impregnated with Solution A containing EtOH and $CaCl_2$ are soaked in 100% water, the sheets are in turn disintegrable because degrees of dissociation of the CMC-COOCaCl salts are enhanced in water. Thus, sufficient wet strengths allow to appear on the sheets impregnated with Solution A in use but the sheets have sufficient disintegrability in toilet water after use.

Fig 8 displays the relationship between DP$_w$ of PTMMAC and the time required for dispersion of the PTMMAC/CMC/pulp sheets to fibers in water by stirring. The disintegrability of the sheets in water sharply decreased or the time required for disintegration of the sheets drastically increased, when the PTMMAC with DP$_w$ of more than 2000 was used in the sheet-making. Thus, the PTMMACs with low DP$_w$ of ~340 are better to be used in sheet-making for the PTMMAC/CMC/pulp sheets having sufficient water-disintegrability after use.

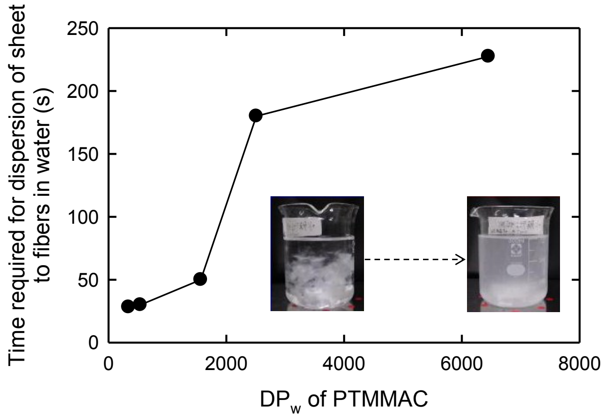

Fig 8. Time required for dispersion of sheets to fibers by stirring in water. The sheets were prepared with 5% PTMMAC and 5% CMC. PTMMACs with different DP$_w$ values were used in sheet-making.

Conclusions

Relatively large amounts of CMC molecules can be retained in the handsheets, when PTMMAC is used as the co-additive in the cationic/anionic dual polymer system. When 5% PTMMAC and 5% CMC are added to the pulp slurry, PTMMAC-N^+ and CMC-COO^- of ~30 mg g^{-1} and 35-38 mg g^{-1}, respectively, were retained in the sheets regardless of DP$_w$ values of PTMMACs. These PTMMAC-N^+ and CMC-COO^- contents in the sheets correspond to ~80% and ~90% retention ratios, respectively; CMC molecules can be retained in the sheets quite effectively by the dual polymer system. Because the surface charges of pulp fibers and colloidal particles in the pulp slurry with 5% PTMMAC and 5% CMC were almost zero, suitable charge balances between anionic and cationic groups in the pulp slurry resulted in such high retention ratios of PTMMAC and CMC in the sheets. The maximum dry tensile strength was obtained for the sheets prepared with 5% PTMMAC and 5% CMC, indicating that the polyion complexes of PTMMAC/CMC electrolytes homogeneously present in the sheets play a role in reinforcing inter-fiber bonds in the sheets. Though the difference in DP$_w$ of PTMMAC between 342 and 6450 had nearly no influence on CMC retention behavior or wet/dry tensile strength, the water-disintegrability of the sheets was significantly affected by the DP$_w$ values of PTMMAC; the lower the DP$_w$, the clearly higher the water-disintegrability of the sheets. Thus, suitable conditions to manufacture the PTMMAC/CMC/pulp sheets for wet wipers can be designed based on the results obtained in this study.

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