

## Deinking of Different Furnishes of Recycled MOW, ONP, and OMG Pulps in Silicate-Free Conditions Using Organic Complex of PHAAS

Iman Akbarpour,<sup>a,\*</sup> Mansour Ghaffari,<sup>b</sup> and Ali Ghasemian<sup>c</sup>

Sodium silicate causes problems in papermaking such as deposit formation, decreased retention, and lower sheet strength. Due to these problems, chemical deinking of different recycled papers furnishes including 100% ONP, 80% ONP, and 20% OMG, and a combination of 70% ONP/ 20% OMG/ 10% MOW was accomplished using an organic complexing agent which included poly-hydroxyl acrylic acid and sodium salt (PHAAS) in silicate-free conditions. PHAAS was utilized at four levels of 0.3, 0.5, 0.7, and 1% (based on oven-dry weight of recycled paper) rather than sodium silicate. The optical and physical properties of deinked pulp were compared to control pulp (conventional deinking containing 2% sodium silicate). The results showed that the paper brightness was improved and the yellowness, dirt count, and dirt area were decreased significantly by increasing PHAAS charge up to 0.9%. Also, in different recycled paper furnishes above mentioned, using different charges of PHAAS had different effects on paper opacity. Using different charges of PHAAS (especially 0.7 to 0.9%) decreased paper caliper, increased paper air resistance, increased freeness, and gave similar or slightly better paper tear indices. Differences of tear indices were not significant at confidence level of 99%. Based on the present research, the use of 0.7 to 0.9% PHAAS in place of sodium silicate is advisable because of the better quality of final papers compared to conventional deinking process.

*Keywords: Chemical deinking; Recycled papers; Organic complex; PHAAS; Optical properties; Physical properties*

*Contact information: a: Ph.D. Student of Pulp and Paper Industries, Gorgan University, Iran; b: M.Sc Graduate of Pulp and Paper Industries, Gorgan University, Iran; c: Associate Prof., Department of Pulp and Paper Industries, Gorgan University, Iran; \*Corresponding author: inakbarpour@gmail.com.*

### INTRODUCTION

Nowadays the shortage of forest-based materials is an important challenge for pulp and paper industry in different countries (Jalal 1989). The pulp and paper manufacturing industries, as the largest consumers of wood, should look for alternative lignocellulosic resources for supplying the raw materials required (Jalal 1989; Akbarpour 2009). At present, recycling of paper as a good solution is attracting more and more attention (Prasad 1993; Bajpai 2010; Pathak *et al.* 2011). The development of technology for pulp and paper manufacture using recycled paper is currently at the center of attention (Park *et al.* 2008; Zanuttini *et al.* 2007; Fatehi *et al.* 2010; Wan *et al.* 2011).

Paper recycling is an effective way to preserve forest resources while saving energy and landfill space. Also papermakers are focusing on recycling as an economic necessity (Borchardt 1993; Cathie and Guest 1998; Ghasemian *et al.* 2006; Pathak *et al.*

2011). Iran has an inadequate supply of natural forest resources that cover only about 7% of the country's land area. Similar to many developing countries, deforestation and over-harvesting in Iran have created environmental issues. On the other hand, increasing demand for forest resources in various applications has led to shortages of wood supply. Therefore, there is a need to look for innovative ways of using non-traditional forest resources to substitute wood raw materials for pulp and paper applications (Ashori *et al.* 2006). The recovered fiber used to make high-grade recycled paper consists mainly of old magazine paper (OMG), mixed office waste (MOW), and old newspaper (ONP) pulps. These recycled furnishes are the main ingredient used for preparing the recycled pulp (Ghasemian *et al.* 2006; Akbarpour 2009).

Deinking is a process for detaching and removing printing inks from recovered fibers. Difficulties in the preparation of the secondary raw material, primarily in separation of ink from cellulose fibres and its removal, are possible in the chemical deinking process. The efficiency of this process depends on the technique and conditions of printing, as well as the kind of ink or printing substrate (Bolanca *et al.* 2000, 2003). The main objective of the deinking process is to produce a good quality paper with good optical and mechanical properties. The chemicals used for deinking recovered papers include sodium hydroxide, hydrogen peroxide, sodium silicate, chelating agents, and surface active agents. Sodium silicate is a buffering agent, peroxide stabilizer, and a softening agent of linkage between printing inks and fibers (McKinney and Hache 1991; Ghasemian *et al.* 2006; Akbarpour 2009). Water streams from the deinking process become contaminated by the numerous chemicals employed and have to be treated.

To overcome the economic and environmental concerns mentioned above, a novel approach employing a single deinking chemical in place of the conventional multi-component mixture formulations was considered. A block copolymer was selected for study. Factors motivating this selection included the possibility of using it in very small concentrations, its good biodegradability properties (some of the block copolymers used are approved by the FDA even as food additives), and the fact that these molecules exhibit a multitude of surface chemical characteristics that are essential for the deinking application (Moon and Nagarajan 1998).

The utilization of a copolymer material has been shown to be useful in combination with surfactants for deinking paper fibers (Nguyen *et al.* 2002). The copolymers of styrene/methacrylic acid function synergistically with various surfactants and provide recycled fiber having characteristics substantially similar to virgin fibers used in papermaking. The concentration of the copolymer component including styrene/methacrylic acid in a deinking solution was preferably between 0.01% and 2% by weight based upon the total weight of the dry fiber to be treated. More preferably, this amount was between 0.04% and 1.5%, with a about 1% being most preferable (Nguyen *et al.* 2002).

The results from using different copolymers in deinking recycled papers including blue security envelopes showed that the use of a water-soluble form of STYMMA 1 (Styrene methacrylic copolymer including 30% styrene/70% methacrylic acid, molecular weight 17,400) is more effective than SURFONIC® (Ethoxylated linear alcohol), as evidenced by the higher brightness value and lower ERIC value. It was unexpectedly found that when STYMMA 1 and SURFONIC®L24-12 are blended, there is a synergistic effect, providing higher brightness values and lower ERIC than either of the components achieve when used alone. Surprisingly, the blend of STYMMA 1 and SURFONIC®L24-

12 as a deinking agent is far superior to that of STYMMA 3 (Styrene methacrylic copolymer including 50% styrene/ 50% methacrylic acid, molecular weight 57,800) and SURFONIC® 124-112. The deinking process of a commercial paper mill producing security envelope, 33.33% ledger stock, and 33.33% book print paper using different copolymers showed that the use of water-soluble form of STYMMA 1 is by itself superior to conventional deinking surfactant, SURFONIC®L24-112 and conventional anti-redeposition agent CMC. Usage increase in these copolymers from 0 to 2% resulted in paper with higher brightness and lower ERIC values. It is preferred that solution employed in a deinking procedure be maintained at a pH level between about 4.0 and 12.0 and it is the most preferred that be about 7.0 to 10.5 (Nguyen *et al.* 2002).

A two-step deinking process involving pulping and flotation steps and utilizing a single block copolymer in very low concentrations, has been developed for xerographic and laser-printed papers. The addition of other chemicals may provide slightly brighter deinked pulp, but their use in a large scale deinking process drives the cost up dramatically. Further, the use of a single chemical offers the opportunity to explore the connection between the surface chemical properties of the additive and its deinking performance.

Poloxamers, which are known commercially as Pluronic (triblock copolymers of the polyethylene oxide (PEO) -polypropylene oxide (PPO) -polyethylene oxide type with the hydrophobic PPO block in the middle attached to the hydrophilic PEO blocks at the two ends) are seen to be effective surfactants for deinking, but the most suitable of these agents should be chosen carefully for a specific application. The poloxamers with the highest PPO molecular weights and the lowest percentage of PEO gave the lowest fiber losses. An ideal poloxamer would provide deinked pulp with a high degree of brightness and little fiber loss. There is a narrow range of poloxamer exhibiting this behavior between the 80 series and the 100 series; the surfactant which seems to be the best in this range is Pluronic L101. The use of this block copolymer also led to very high ink removal efficiencies, as determined using computerized image analysis (Moon and Nagarajan 1998).

The use of organic polymers as stabilizers in peroxide bleaching has been studied since the 1950's. Polyvinylpyrrolidone maleic acid-styrene or propenes, co-polymers of butadiene-maleic acid, and grafted polymers are examples of polymeric stabilizers that have been reported in the literature. Hydroxyacrylate in peroxide bleaching has been reported to be an effective stabilizer for peroxide bleaching (Ni 2005; Wurorimaa *et al.* 2006). The results show that silicate and PHAS have the highest peroxide stabilizing efficiency in terms of brightness and residual peroxide (Hamalainen *et al.* 2007). Polyhydroxyl acrylic acid is an effective stabilizer due to its metal binding ability and possibly due to its radical scavenging ability. In many cases it is advantageous to apply PHAS together with a calcium-binding polymer (Leduc *et al.* 2005; Hamalainen *et al.* 2007).

MA/AA and polyacrylate failed to show proper stabilization effect. Recently, it was proposed to use organic complexes in place of silicate to control of scaling in equipment in virgin pulp bleaching and paper recycling (Wuorimaa *et al.* 2006; Hamalainen *et al.* 2007). The results from using different chemicals in place of sodium silicate in birch CTMP bleaching in basic alkaline conditions showed that an organic complex of poly-acrylic acid and mineral salts (trade name: Paf) produced the best alternative for sodium silicate as compared to other chemicals applied. Also, by using a

dosage of 0.8%, a brightness of 68.7% was achieved in alkaline peroxide bleaching. This value was more than that of the paper brightness in conventional bleaching process using 2% silicate (Dehghani 2004). The results show that paper with similar or higher brightness and lower yellowness could be produced in silicate-free or silicate-containing peroxide bleaching of CMP pulp using the sodium salt of poly-phosphonic acid. When silicate was eliminated from peroxide bleaching chemicals along with substitution by this complex, the highest brightness (65.5% ISO) and the lowest yellowness (21.6% ISO) were observed. With an increase in complex charge up to 0.5% combined with 0.7% silicate, the brightness was improved by up to 68.1% ISO, and the yellowness decreased to 20.8. At higher concentrations (more than 0.5%) complex, the brightness was increased and the yellowness decreased. By use of this complex in place of sodium silicate, it was possible to better manage the peroxide bleaching effluent in terms of scale control, effluent recycling, and chemical recovery (Akbarpour and Resalati 2008). The effects of a non-silicate organic stabilizer derived from poly-hydroxylacrylic acid and sodium salt (PHAAS) was investigated on bleached pulp brightness and yellowness in peroxide bleaching of mill made with mixed hardwood CMP pulp. It was found that, in conventional single-stage peroxide bleaching with silicate, the highest brightness and the lowest yellowness obtained were 66.8% and 22%, respectively, at 3% silicate. On the other hand, the highest brightness (66.1%) and the lowest yellowness (20.5%) were observed using 0.5% PHAAS, which were very comparable with silicate bleaching. At lower or higher charges than 0.5% PHAAS, a decrease in brightness and an increase in yellowness was observed. However, when a mixture of 0.5% PHAAS and 0.7 to 1% silicate was used in peroxide bleaching stage, the optical properties were further improved (Akbarpour and Resalati 2008).

Silicate may generate serious detrimental effects in the pulp and paper processes. Silicate compounds may build up in the papermaking process, cause deposits on the paper machine, and introduce anionicity into a paper making system, leading to higher wet-end chemicals consumption (Yonghao 2005; Hamalainen *et al.* 2007). There are good prospects for the use of silicate-free peroxide stabilizers in order to get rid of the aforementioned problems. On the other hand, silicate has its own problems of stability, and it causes fouling of equipment, coating of fibers, harshness of paper, *etc.*, due to the formation of silicate deposits. Efforts have been oriented on finding an effective polymeric stabilizer for sodium silicate. There have not been enough studies in terms of replacing the organic complexes to silicate in deinking of recovered paper. In the present research, the sodium salt of poly-hydroxy acrylic acid (PHAAS, commercially known as Sulvy-x) was applied at different levels. The effect of different charges of PHAAS was investigated on the optical and physical properties of paper produced. Finally, the results were compared with those of control pulp (deinked pulps using conventional method in the presence of 2% sodium silicate).

## EXPERIMENTAL

### Preparation and Repulping of Recovered Papers

The recovered papers used in this work were ONP, MOW, and OMG, all of which were collected from a local company. The initial brightness, yellowness, and opacity of undeinked ONP, MOW, and OMG pulps are shown in Table 1.

**Table 1.** Optical Properties of the Recovered Fibers as Received

Pulp type	ONP	MOW	OMG
Brightness (%)	44.3	79.8	41.2
Yellowness (%)	10.4	8.43	11.74
Opacity (%)	98.9	86.9	96.2

### Chemical Pulping of Recovered Papers

Different furnishes of recovered papers (mixtures of 100% ONP, 80% ONP/20% OMG, and 70% ONP/20% OMG/10% MOW) were disintegrated using tap water for 10 min in a laboratory pulper operated at pulp consistency of 5% and a temperature of 50 °C. The chemicals added in pulping were sodium hydroxide, hydrogen peroxide, chelating agent, sodium silicate, and other collector chemicals according to Table 2. All chemicals were added based on oven-dry paper basis. Pulper pH was adjusted to 10.5 to 11.5. In this research, the control samples were prepared from pulping of different furnishes with conventional chemicals corresponding to Table 1. The other treatments were done in silicate-free conditions by substitution of different charges of PHAAS complex.

**Table 2.** Chemical Treatment Conditions for Recycling of Recovered Papers

Chemicals Type	Consumption (Based on oven-dried weight of pulp) %
Hydrogen peroxide	1
Sodium hydroxide	1
DTPA	0.3
Surfactant (Tween 80)	0.15
Sodium silicate	2

### Organic Complex of PHAAS

Organic complexes are used as a fixing or stabilizing agents in the hydrogen peroxide bleaching and deinking of recovered papers (Akbarpour and Resalati 2008). Organic complex derived from poly-hydroxyl acrylic acid and sodium salt (PHAAS) was prepared. In mills, it is made generally with a concentration of 10%. PHAAS was obtained as a liquid form which had a bright yellow color and was slightly odorous. The physical and chemical characteristics of PHAAS are given in Table 3. At the constant conditions mentioned above, the organic complexing agent of PHAAS was used at different charges of 0.3%, 0.5%, 0.7%, and 0.9% (based on oven-dried weight of recycled paper) combined with deinking chemicals without sodium silicate.

**Table 3.** The Physical and Chemical Characteristics of PHAAS

pH (at 20 °C)	8-9
Boiling point (°C)	100
Relative density (at 20 °C)	1.26-1.3
Water solubility	Completely soluble
Flash point	Not applicable

### Washing and Flotation Deinking

The resultant pulps from different treatments were washed with tap water through a 200-mesh wire screen and subjected to flotation (capacity of 20 litres equipped with an aerator operating at speed of 1300 rpm with the airflow set to 10 L/min) for 20 min at a temperature of 40 to 50 °C and a consistency of 0.8 % in the presence of surfactant (0.15% Tween 80) and (0.3% CaCl<sub>2</sub>) as flotation aids. During flotation, foam with ink particles attached was continually skimmed away from the cell surface. In all furnishes, a washing stage was implemented before flotation in order to increase brightness, to decrease yellowness, opacity, dirt count, dirt area, and caliper, and also to improve the pulp freeness when compared to undeinked pulp (data not shown). After flotation, the pulps were prepared to make handsheets.

### Determination of Handsheet Properties

The handsheets (60 gsm) were made according to TAPPI T205sp-95. The optical and physical properties of papers were measured according to TAPPI test methods as follows: brightness and yellowness, TAPPI T452-om-02; opacity, TAPPI T425 om-02; pulp freeness (mL, CSF), TAPPI T227 om-04; caliper, TAPPI T411 om-05; air resistance, TAPPI T 460 om-02; tear resistance, TAPPI T414 om-04. Also, the paper density was measured at different treatments with relation between caliper and basis weight of papers. Finally, the optical and physical properties of papers were compared at different charges of PHAAS complex to control pulp (different recovered papers deinked with chemicals mentioned in Table 2).

### Measurement of Dirt Count and Dirt Area

The image analysis of the handsheet was carried out using a computer notebook, an Epson Perfection V200 Photo scanner, and Spec San 2000 software version V.2.3.30. The scanning condition used was according to the TAPPI Dirt Estimation Chart setting (TAPPI T537 om-96). Dirt count (number) and dirt area (mm<sup>2</sup>) per sheet area (m<sup>2</sup>) were reported. It should be mentioned that the lower limit of the ink particle size to be detected was 0.04 mm<sup>2</sup>.

## RESULTS AND DISCUSSION

According to variance analysis, different charges of PHAAS in silicate-free condition to deink mixed recycled papers were found to have a significant effect on brightness when compared to the control pulp. Increasing the PHAAS consumption charge from 0.3% to 0.5% did not have a significant effect on the brightness values of produced paper. However, a further significant effect was observed when the PHAAS dosage was increased to 0.9%. Using 0.9% PHAAS, the highest brightness was achieved, reaching the same level as the control pulp. Mean values analysis (Duncan) classified these values into separate groups. In general, the highest levels of PHAAS resulted in paper with higher brightness (Fig. 1). Variance analysis of yellowness values indicated significant effects at different levels of PHAAS, at confidence levels 99%. Duncan analysis classified the yellowness value into separate groups (Fig. 2). Hydroxyl-acrylate in peroxide bleaching has been reported to be an effective stabilizer in peroxide bleaching (Yonghao 2005; Wurorimaa *et al.* 2006). Poly-hydroxyl acrylic acid is an effective

stabilizer due to its metal binding ability and possibly due to its radical scavenging ability. In many cases, it is advantageous to apply PHAS together with a calcium-binding polymer (Leduc *et al.* 2005; Hamalainen *et al.* 2007). Also, it was reported that PHAS components have the highest peroxide stabilizing efficiency in terms of brightness (Hamalainen *et al.* 2007). It seems that peroxide has a similar function in paper recycling. So, improvement of brightness and decrease of yellowness are attributed to the bleaching function of peroxide. Optimum usages of PHAAS were different in all recycled paper furnishes applied. In general, by addition of PHAAS up to 0.9%, better brightness and lower or similar yellowness were achieved. It should be mentioned that the optical properties of the produced paper were decreased at higher charges than 0.9 percent of PHAAS (data not shown).

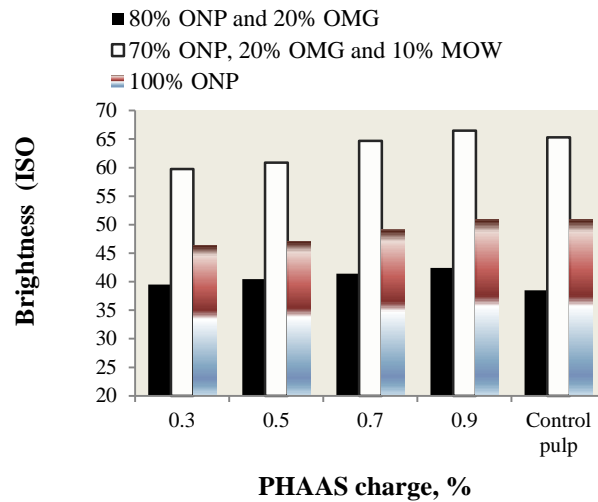


Fig. 1. The effect of different PHAAS charges on paper brightness

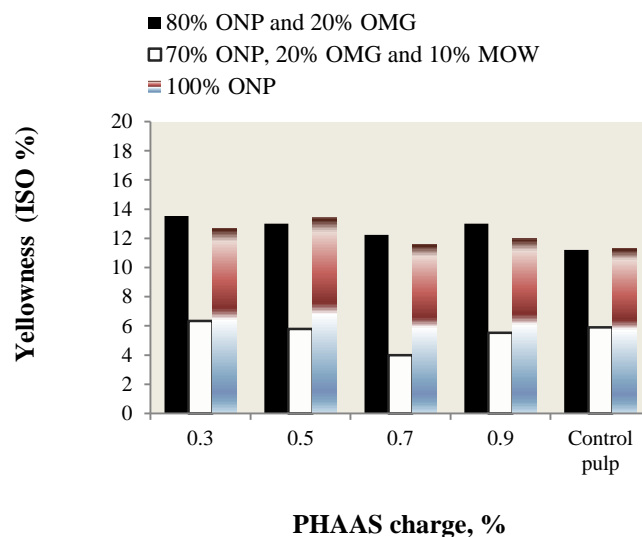
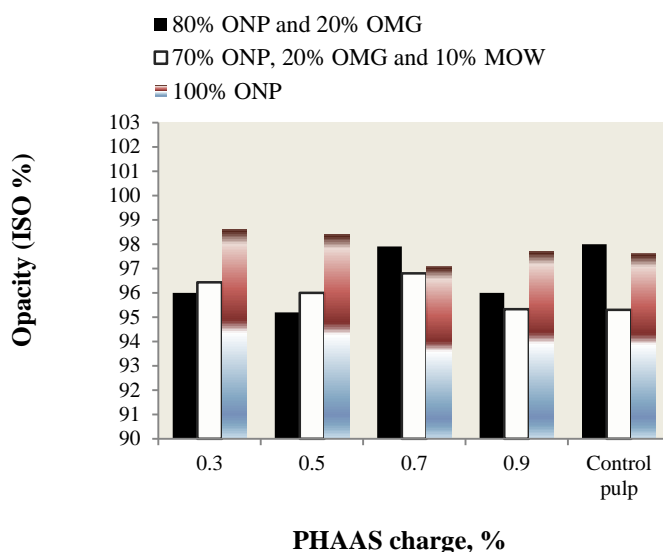


Fig. 2. The effect of different PHAAS charges on paper yellowness

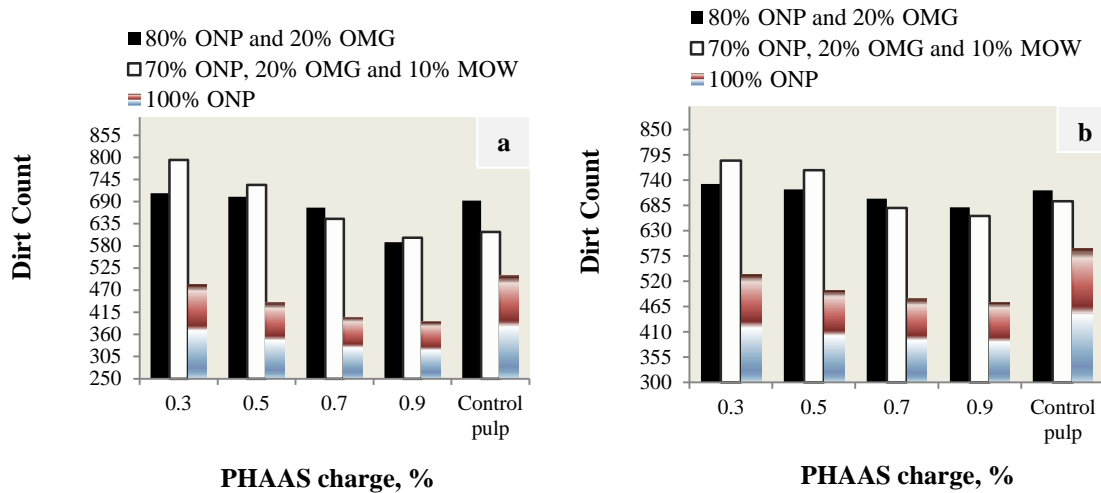
Variance analysis of the opacity values showed a significant effect between treatments in terms of different charges of PHAAS, compared to the control pulp. In general, using different PHAAS charges rather than sodium silicate generally resulted in paper with similar or even more opacity in some furnishes of pulp. Different furnishes of recycled papers showed different opacity values because of the different types of pulp and or printing inks present in stock applied. So that the highest opacity achieved were different in any furnishes of recycled papers (Fig. 3). Increasing or decreasing of the opacity could be attributed to increase or decrease of bonding capacity in recycled fibers. In principle, when inter-fiber bonding is reduced, it can be expected that the light scattering and opacity of papers will be increased.



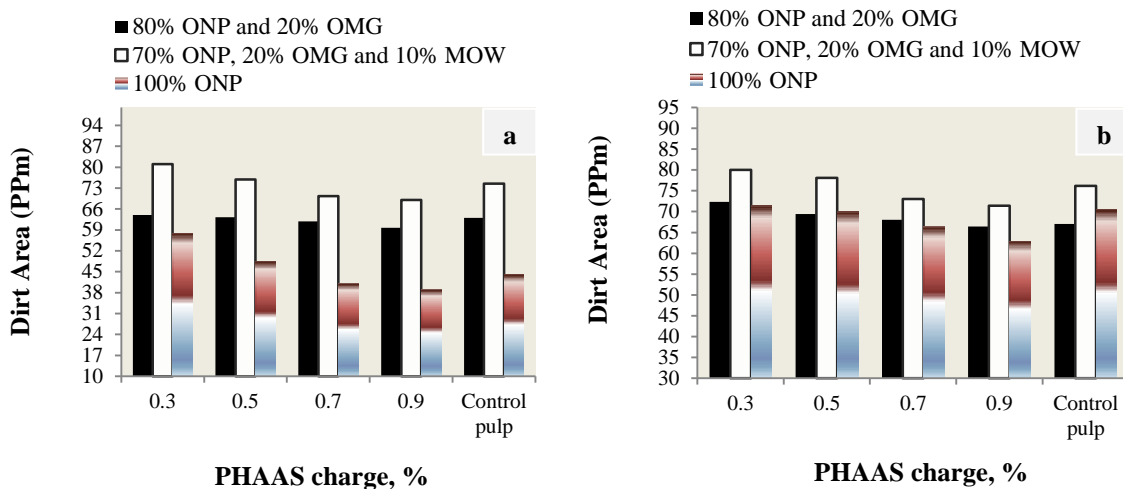
**Fig. 3.** The effect of different PHAAS charges on paper opacity

The results obtained from comparing the ERIC values, which included dirt count and dirt area of handsheets in double side, showed that replacement of the silicate with PHAAS resulted in a significant improvement in ink removal percentage relative to the control samples ( $P < 0.01$ ). When PHAAS charge was increased, the ERIC values of handsheets were decreased (Figs. 4 and 5). These differences of ERIC values were significant ( $P < 0.01$ ). It could be stated that the complex of PHAAS containing carboxylic acid groups is hydrophilic and has higher surface active and different chemical properties than another chemicals found in conventional deinking. It doesn't agglomerate ink particles but rather disperses the ink particles, thus minimizing their tendency to become re-deposited on the fibers. According to this research, the concentration of the PHAAS component in deinking of different furnishes of recycled paper before mentioned is preferably between 0.7% and 0.9% by weight based upon the total weight of the dry fiber. The final papers had higher brightness and lower dirt count and dirt area values. The improvement of ink removal was slightly greater in mixed MOW, ONP, and OMG furnish than the other furnishes.





**Fig. 4.** The effect of different PHAAS charges on paper dirt count, a (Wire side), b (Felt site)



**Fig. 5.** The effect of different PHAAS charges on paper dirt area, a (Wire side), b (Felt site)

The results from variance analysis of freeness values (mL, CSF) at different charges of PHAAS indicated that there were significant differences between various charges of PHAAS at a confidence level of 99%. There was different results in Duncan grouping from different furnishes applied. Overall, using 0.7% PHAAS yielded in a pulp with average higher freeness or better drainage properties (Fig. 6).

Using different levels of PHAAS instead of sodium silicate resulted in a significant decrease of paper caliper compared to that of the control pulp. Based on the Duncan analysis, there was not a significant effect on caliper when PHAAS charge was increased beyond 0.7%. In general, the lowest caliper (Fig. 7), highest density (Fig. 8), and highest air resistance (Fig. 9) were related to 0.7 or 0.9% PHAAS as compared to the control pulp. With regard to achieved results, higher charges of PHAAS resulted in papers having higher density and greater air resistance.

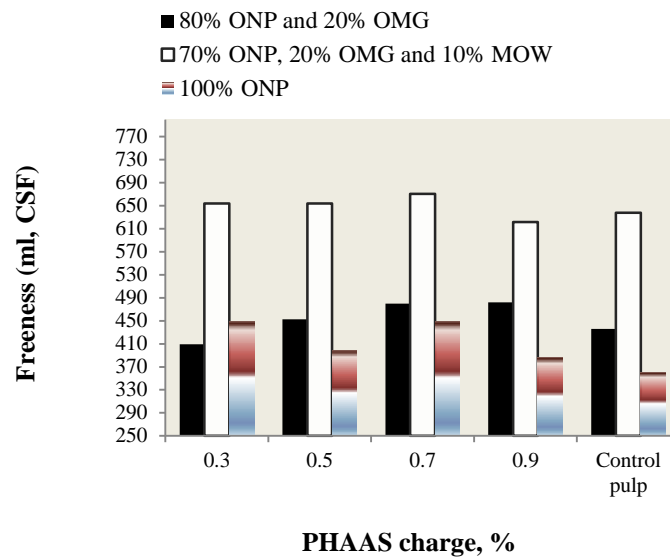


Fig. 6. The effect of different PHAAS charges on pulp freeness

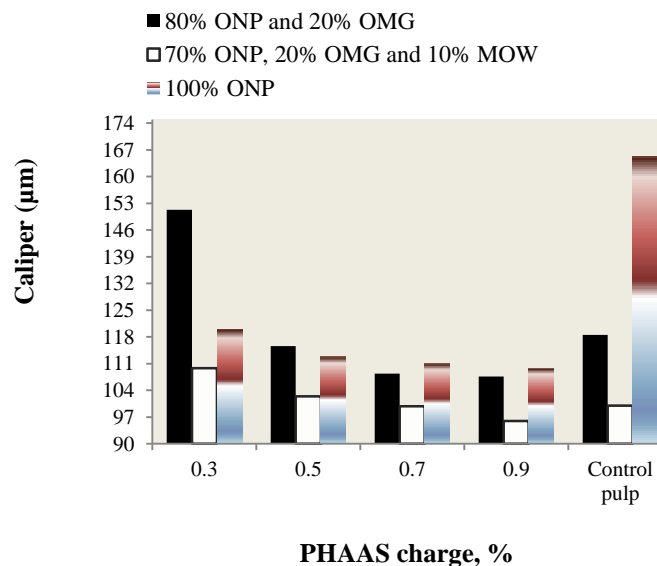
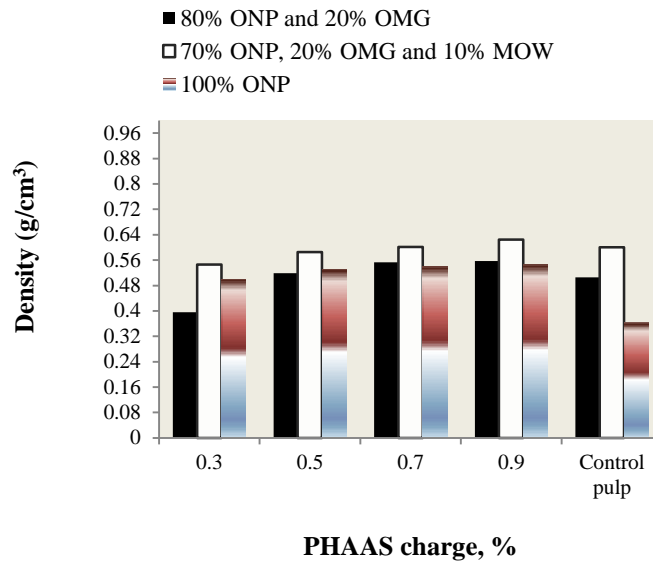


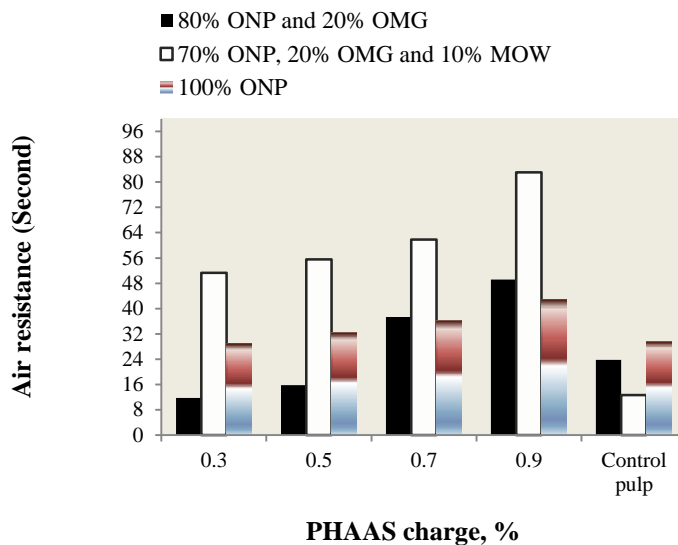
Fig. 7. The effect of different PHAAS charges on paper caliper

Bulk and density are more important properties affecting the physical, mechanical, electrical, and printability properties of paper. The observed enhancement of the paper caliper is attributed to reduced fiber bonding or bonding capability, leading to reduced apparent density. Use of the organic complexing agent PHAAS improved the fiber bonding and resulted in a paper with more density. Higher charges of PHAAS (0.7%) resulted in paper having higher density and lower caliper. Although the carboxylic groups content in the pulps treated with PHAAS were not analysed, it is expected that a suitable reason for this observation can be the number of cellulose carboxyl groups present in pulps, which was increased by increases in PHAAS. Therefore, the caliper of

paper is reduced and air resistance is enhanced by increasing of hydrogen bonding between fibers. Meanwhile, carboxyl groups present in the chemical structure of PHAAS could increase hydrogen bonding, which contributes to fiber bonding. Fibres with a higher carboxylic acid groups swell more, exposing larger surface areas with more potentially reactive sites available for interaction (Eriksson and Sjöström 1968). The increase in fibre flexibility and conformability increases the bonding of fibres and decreases the paper caliper (Laine 1996).



**Fig. 8.** The effect of different PHAAS charges on paper density



**Fig. 9.** The effect of different PHAAS charges on paper air resistance

As shown in Fig. 10, there was not a significant effect on tear indices of papers corresponding to variance analysis. In general, the results showed that one could produce a paper with similar tear index or even higher than the control pulp using different charges of PHAAS from deinking of different furnishes of recycled papers (Fig. 10). The use of 0.7 to 0.9% PHAAS in all experiments in different furnishes resulted in paper with higher tear indices.

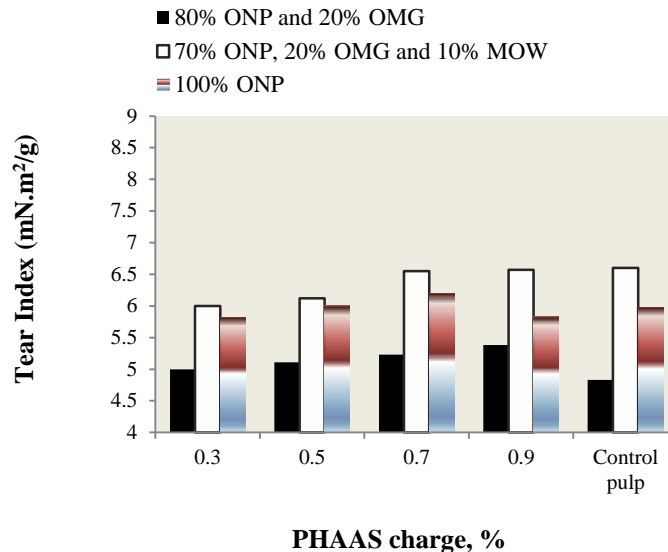


Fig. 10. The effect of different PHAAS charges on paper tear index

## CONCLUSIONS

Silicate compounds may build up in the papermaking process, causing deposits on the paper machine and decreasing the paper strength and higher consumption of wet-end chemicals. In this study, silicate-free deinking of different recovered papers was evaluated, and the results were as follows:

1. It was demonstrated that silicate-free conditions can be used successfully for deinking of recovered papers including ONP, MOW, OMG, or combinations of them using organic complex of poly-hydroxyl acrylic acid and sodium (PHAAS) as an alternative replacement to sodium silicate in deinking formulations. The present results showed that it is possible to produce the papers with suitable quality compared to both conventional deinked pulps.
2. Addition of PHAAS rather than sodium silicate improved the paper brightness, air resistance, paper density, pulp freeness and decrease of paper yellowness compared to different respected control pulps which deinked conventionally with presence of 2% sodium silicate. By replacement of PHAAS, there was not found to be any significant difference between tear indices when compared to control pulp and obtained tear indices of paper produced with different charges of PHAAS were comparable and sometimes higher than those of the control pulps.

3. The furnishes ONP, MOW, and OMG have basically different chemical and mechanical characteristics such as pulp type, ink, and printing types. So it is imagined they have a different response to the deinking treatments. In this research there were different results from different levels added of PHAAS to deinking chemicals. Overall using 0.7 to 0.9% PHAAS resulted in better optical and physical properties and it is suggested for deinking of these furnishes.
4. With regard to major problems of silicates in terms of their build-up in papermaking process, deposit formation and wastewater treatment cost, it could be expected that using PHAAS is better as economic point of view compared to sodium silicate.

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