EFFECT OF PARAFFIN WAX CHARACTERISTICS ON THE FORMULATION AND RHEOLOGY OF WAX-BASED BINDERS

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ABSTRACT

The effect of paraffin waxes on the formulation characteristics and rheology of wax-based binders have been examined. Four paraffin waxes (PW_1, PW_2, PW_3 & PW_4) were selected and compared with the foreign paraffin wax (PW_5). Ten binder systems were formulated by mixing the major binder consisting of the paraffin wax with or without stearic acid (SA) with the minor binder; poly (ethylene-co-vinyl acetate) (EVA). The binder systems 1-4 and 6-10 were compared with the well known one no.5 (which contains PW_5). The physical characteristics and molecular type composition of the paraffin waxes were studied. Differential scanning calorimetry (DSC) and thermo gravimetric analysis (TGA) were used to evaluate the thermal characteristics of the pure binder components and of the formulated binder systems. Finally, the rheological properties of the formulated binder systems were studied to measure their viscosities. Data indicated that the binder system no. 4 consisted of 35 wt. % EVA (containing vinyl acetate content of 9 wt %), 62 wt. % of PW_4 and 3 wt % of SA is the preferable binder for powder injection molding.

Keywords: Paraffin waxes; formulation characteristics; wax-based binders; thermal characteristics; rheological properties.

1. INTRODUCTION

Powder injection molding (PIM) is an advanced technology for processing metal and ceramic powders for forming desired shapes. PIM process combines the plastic injection technology and the conventional powder technology for sintering molded parts. Therefore it is capable of producing a wide range of high performance and complex shaped parts at a relatively low processing cost [Pease, (1987), Hsu et al., (1996) & German, (1993, 2003)]. The PIM consists of the formulation of a binder, the preparation of feedstock by mixing of binder and metal or ceramic powders, injection molding of the feedstock into a mold, removal of the binder from the feedstock (solvent and thermal debinding), and sintering of the metal or ceramic powders [Park & Kim, (2001) & Zaky, (2004)].

The selection of proper binder is a prerequisite for producing complete PIM parts successfully [Hsu et al., (1996)]. The role of the binder is to serve as a temporary or transient phase to impart flowability and moldability. This will enable the shapping of the feedstock to the required shape during injection molding [Loh et al., (2001)].

An ideal binder system for PIM should have superior attributes: flow characteristics, interaction with powder, debinding and manufacturing. Thus, the binder should have viscosity below 10 Pa.s at the molding temperature, low viscosity change with temperature during molding and rapid viscosity change during cooling. The binder should have low contact angle and adhere well to the powder during molding. During debinding, the binder should hold the shape and be totally decomposed before sintering with low ash content. For manufacturing, the binder should be inexpensive and environmental friendly [Chung et al., (1989); German and Bose, (1997); Liu et al., (2001); Goncalves, (2001)].

To fulfil the above requirements a successful binder for the PIM process requires a multi-component mixture. Common binders in PIM are wax-based binder systems. When heated, these binders provide a liquid phase for transporting the powders into the mold cavity. At the end of the molding cycle, the cooled binder freezes to provide a sufficient strength to resist damage during injection [German, (2003)]. In the wax-based binder system, the common backbone polymers are polyethylene and polypropylene. Various waxes and acids are also added to the binder

to lower down the viscosity and increase the wettability and miscibility [Qingfa et al., (2002)].

A favorite binder system is composed of 65% paraffin wax, 30% polypropylene and 5% stearic acid [German, (2003) & Kim et al., (1999)] used a wax-based binder system consisting of paraffin wax, ethylene vinyl acetate copolymer (EVA), polyethylene wax (PE wax) and stearic acid. They investigated the effect of the type of minor binder (polyethylene wax or EVA) on the removal process of major binder (paraffin wax) and the pore structure evolution of partially debinded body in the course of the wicking of compression molded parts. Attarian et al., (2002) used a binder system consisting of polyethylene wax as a major binder, varying amount of EVA as a minor binder and stearic acid as a constant component. They studied the influence of EVA copolymer content on the rheological behavior of alumino-silicate / polyethylene wax suspensions. Li et al., (2003) used a new wax-oil-polyethylene binder. They added the oil into the wax-polyethylene binder in order to secure a quickly solvent-removable binder. Hsu et al., (1996) compared the effect of different waxes (paraffin wax, polyethylene wax, carnauba wax and acra wax) on the injection molding of carbonyl iron powder with low density polyethylene. Kim et al., (2000) used a binder system consisting of 65 % paraffin wax purchased from Oxford Co. (St. Louis, Mo.), 32 % poly (ethylene-co-vinyl acetate) (EVA), and 3 % stearic acid.

Thus, the aim of our study is to compare the effect of some Egyptian paraffin waxes having different characteristics and compositions on the formulation characteristics and rheology of wax-based binders.

2. EXPERIMENTAL

2.1. Materials

Poly (ethylene-co-vinyl acetate) (EVA) containing 9 and 9.5 wt. % vinyl acetate were used as a minor binder component. The former was purchased from Aldrich Co., while the latter was kindly supplied by Hanwha Chemical Co. (Toejon, South Korea).

Light (PW_1), middle (PW_3) and heavy (PW_4) paraffin waxes were separated from three local slack waxes from El-Ameria Refining Company. PW_2 is a mixture of 60 wt. % PW_1 and 40 wt. % PW_3.

Foreign paraffin wax (PW_5) was purchased from Oxford Co. (St. Louis, MO). Each of these paraffin waxes was used as the major binder component.

2.2. Binder System Formulations

Ten wax-based binder systems were prepared by mixing the minor binder and the major binder (with or without stearic acid) using a mechanical stirrer at 130 °C for 30 min. They are as follow:

1) 35 wt. % EVA, 62 wt. % PW_1 and 3 wt. % stearic acid.
2) 35 wt. % EVA, 62 wt. % PW_2 and 3 wt. % stearic acid.
3) 35 wt. % EVA, 62 wt. % PW_3 and 3 wt. % stearic acid.
4) 35 wt. % EVA, 62 wt. % PW_4 and 3 wt. % stearic acid.
5) 35 wt. % EVA, 62 wt. % PW_5 and 3 wt. % stearic acid.
6) 35 wt. % EVA and 65 wt. % PW_1.
7) 35 wt. % EVA and 65 wt. % PW_2.
8) 35 wt. % EVA and 65 wt. % PW_3.
9) 35 wt. % EVA and 65 wt. % PW_4.
10) 35 wt. % EVA and 65 wt. % PW_5.

The binder systems 1-4 and 6-10 were compared with the well known binder system no. 5 [Kim et al., (2000)], which consists of 35 wt. % EVA containing vinyl acetate content of 9.5 wt. %, 62 wt. % of foreign paraffin wax (PW_5) and 3 wt. % of stearic acid (SA).

2.3. Methods of Analysis

2.3.1. Physical Properties

The isolated paraffin waxes were physically characterized according to ASTM standard methods [ASTM, (1995 & 1999)]. The standard methods for analysis are as follows:

Congealing Point	ASTM D-938-98
Kinematic Viscosity	ASTM D-445-97
Refractive Index	ASTM D-1747-94
Mean Molecular Weight	ASTM D-2502-92
Density	ASTM D-1418-99
Oil Content	ASTM D-721-97

Needle Penetration   ASTM D-1321-97
Color   ASTM D-1500-98
Sulfur content, X-ray Fluorescence sulfur meter   ASTM D-4294-90
Carbon number   ASTM D-5442-93

2.3.2. Molecular Properties

Infrared spectra of the studied paraffin wax samples were recorded via FTIR spectrophotometer (Model 960 Mooog, ATl Mattson Infinity Series, USA). The samples were analyzed using KBr cells to determine their crystallinities and branching indices. The spectra of all studied paraffin wax samples were measured in the range of 4000 – 400 cm^{-1} by summing 32 scans at 4 cm^{-1} resolution and 32 background scans. Some ratios calculated from the peak heights of selected infrared bands allow a better study of the spectra. The ratio of absorbance of CH_2+CH_3 asymmetric deformation at 1470 cm^{-1} to that of CH_3 symmetric deformation at 1380 cm^{-1} (A_{1470} / A_{1380}) is a measure of chain length and / or branching index [**Blanco et al., (1996)**]. The crystallinity which is the ratio of CH_2 rocking bands at 730 and 720 cm^{-1} has been determined when both bands are found [**Srivastava et al., (1992)**].

The aromatic content of each of the isolated paraffin waxes was determined using liquid-solid column chromatography technique [**Snyder, (1975)**].

n-Paraffin content was determined for the isolated paraffin waxes using GC technique. The GC apparatus used was model 6890 plus Aglient, equipped with a hydrogen flame ionization detector and fused silica capillary column (30 m x 0.25 mm i.d.), packed with poly (dimethyl siloxane) HP-1 (non-polar packing) of 0.5 μm film thickness. The peak area of each resolved component (consisting of n- and iso-paraffins) is determined individually. However, the unresolved complex mixture (hump); composed of non n-paraffins presumably iso- and cyclo-paraffins and aromatics with long side chains; is determined only as a total.

Ultraviolet absorbance for the isolated paraffin waxes was determined at 290 nm by using UV-Visible Spectrophotometer.

Differential scanning calorimetry (DSC) data are valuable for powder injection or compression molding in providing the general mixing and molding temperatures. Thus, a Setaram instrument of Lab-

system TG-DSC model 16 coupled with computer unit was used to test the thermal properties of the binder components (paraffin waxes, EVA and SA) and the binder systems in the temperature range of 25 to 250 ^{0}C. A heating rate of 10 ^{0}C / min was used and the purge gas used was argon.

Thermo gravimetric analysis (TGA) was used to obtain the thermal stability of pure binder components. It was conducted on a Shimadzu TGA-50. The pyrolysis of binder components was conducted in temperature range of 30 to 600 ^{0}C, under dynamic nitrogen gas. The heating rate used was15 ^{0}C/min.

The most important parameter in rheological measurements is the viscosity. Thus, a Brookfield Digital Rheometer of HBDV-III+ model with spindle 52 was used to measure the viscosity of the binder systems at different temperatures ranging from 120 to 140 ^{0}C.

3. RESULTS AND DISCUSSION

3.1. Physical Characteristics

The physical characteristics and molecular type composition of the four separated paraffin waxes (PW_1-PW_4) and the foreign one (PW_5) are summarized in Tables 1 and 2, respectively.

Data in Table 1 indicate that the congealing point, viscosity, refractive index and density for the four separated paraffin waxes increase with increasing the boiling point range and the mean molecular weight of the paraffin wax, PW_1, PW_3 and PW_4 separated from light, middle and heavy slack waxes, respectively.

In spite of the highest content of iso- and cyclo-paraffins and highest ratio of iso- and cyclo-paraffins to n-paraffins for the paraffin wax PW_4 (Table 2). its congealing point is the highest value and needle penetration is the lowest. This is attributed to the highest boiling point range of the wax distillate fraction from which the heavy slack wax is derived. Carbon number analysis and mean molecular weight data are in line with the previous findings as the paraffin wax PW_4 has the highest mean molecular weight (521) and carbon chain number (C_{23}-C_{43}) as compared with the other separated paraffin waxes.

Table (1): Physical characteristics of pure binder components.

Characteristics	Pure binder components						
	PW₁	PW₂	PW₃	PW₄	PW₅	SA	EVA
Congealing point, °C	53	58	63.5	69	63	----	----
Melting point, T_m °C (DSC)	----	----	----	----	----	62	100.5
Kinematic viscosity, 98.9 °C, cSt	2.88	3.31	3.95	5.26	5.00	----	----
Refractive index, 98.9 °C	1.4186	1.4207	1.4232	1.4253	1.4231	----	----
Density, 20 °C, g/cm³	0.8645	0.8689	0.8852	0.9065	0.900	0.900	0.930
Mean molecular weight	378	406	448	521	500	----	----
Mean molecular weight (GPC)	----	----	----	----	560	----	74,800
Oil content, wt.%	0.45	0.22	0.15	0.14	0.15	----	----
Sulfur content, wt. %	0.00	0.00	0.00	0.00	0.00	----	----
Needle penetration, 25 °C	22	17	15	14	15	----	----
Color (ASTM-D 1500)	0.0	0.0	0.0	0.0	0.0	----	----
UV absorbance @ 290 nm	0.00	0.00	0.04	0.00	0.00	----	----

Table (2): Molecular type composition of paraffin waxes.

Molecular Type Composition	Paraffin Waxes				
	PW₁	PW₂	PW₃	PW₄	PW₅
Total saturates, wt %	100	100	100	100	100
n-Paraffins content, wt %	88.14	84.77	79.73	68.67	76.05
Iso & cycloparaffins content, wt %	11.86	15.23	20.27	31.33	23.95
Iso & cycloparaffins / n-paraffins ratio	0.13	0.18	0.25	0.46	0.31
Total aromatics, wt %	0.00	0.00	0.00	0.00	0.00
Carbon number analysis	C_{19}-C_{33}	C_{20}-C_{34}	C_{23}-C_{35}	C_{23}-C_{43}	C_{21}-C_{42}
Crystallinity, % (A_{730}/A_{720} x 100)	93.55	94.66	97.10	97.05	97.50
Branching index (A_{1470}/A_{1380})	2.21	2.23	2.50	5.46	5.39

3.2. Molecular Type Composition

Data in Table 2 show that all the separated paraffin waxes have saturate contents of 100 %, i.e.. have no aromatic content. This means that, they are classified as fully refined food grade (pure) paraffin waxes [Sequeria, (1994)] as they are white in color. their oil contents less than

0.5 wt. % and their ultraviolet absorbance at 290 nm less than 0.12 (Table 1).

It can be noticed from FTIR data that, the crystallinity and branching index increase also by increasing the boiling point range and/or the mean molecular weight of the paraffin wax. i.e., in the following sequence: $PW_1 < PW_2 < PW_3 < PW_4$ (Table 2).

Comparing the physical characteristics and molecular type composition of the four separated paraffin waxes with those of the foreign one, it can be noticed that PW_1 and PW_2 show different characteristics and compositions from the foreign one PW_5. PW_3 has nearly the same congealing point. refractive index, n-paraffin content and crystallinity as PW_5. Meanwhile the viscosity, density. mean molecular weight and carbon chain number are lower than those of PW_5. The congealing point and refractive index of PW_4 are higher and its n-paraffins content is lower than those obtained for PW_5 whereas, its viscosity. density. mean molecular weight. carbon chain number, branching index and crystallinity are nearly the same as PW_5 (Tables 1 & 2).

3.3. Differential Scanning Calorimetry

The results obtained from DSC measurements for the binder components (PW_1, PW_3, PW_4, PW_5, SA & EVA) and the formulated binder systems are summarized in Tables 3 and 4, respectively. Figures 1 & 2 show the DSC heating curves of the binder components and the formulated binder systems respectively. Two endothermic peaks are shown for each heating curve for the paraffin waxes (PW_1, PW_3, PW_4 & PW_5) and EVA polymer represent the solid-solid and solid-liquid transition temperatures of the material. While the heating curve of SA exhibits one endothermic peak which represents the solid-liquid transition temperature (Fig. 1). The solid-liquid transition temperature represents the melting point of the material.

Table (3): Parameters obtained from DSC measurements for the pure binder components.

Sample	Peak Temperatures, T_m, ^{0}C		Congealing Point, ^{0}C
	Solid-solid transition	Solid-liquid transition	
PW_1	37.47	55.37	53
PW_2	----	----	58
PW_3	48.96	64.63	63.5
PW_4	54.71	69	69
PW_5	45.16	63.08	63
SA	----	62.14	----
EVA	61.32	100.53	----

Table (4): Parameters obtained from DSC measurements for the binder systems.

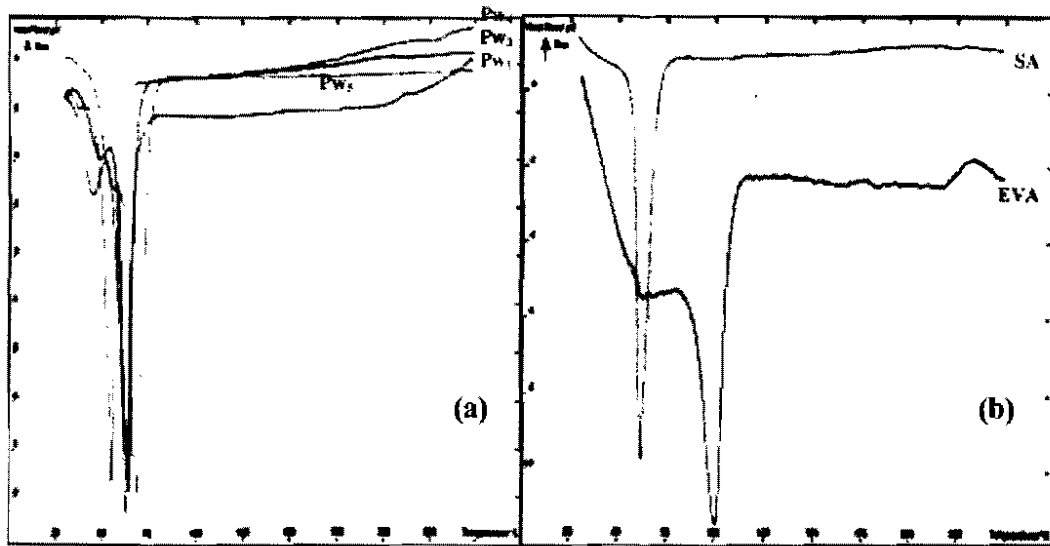| Sample	Peak Temperatures, T_m, ^{0}C		
	Solid-solid transition	Melting Points, ^{0}C	
		Wax	Polymer
Binder 1	-----	58.70	86.62
Binder 2	-----	58.47	86.07
Binder 3	47.37	63.98	85.71
Binder 4	-----	69.95	88.49
Binder 5	-----	62.63	86.96
Binder 8	50.56	68.76	88.42
Binder 9	-----	69.97	86.21
Binder 10	-----	63.80	87.99

Fig. 1: DSC curves of pure binder components (a) PW_1, PW_3, PW_4 & PW_5 and (b) SA & EVA.
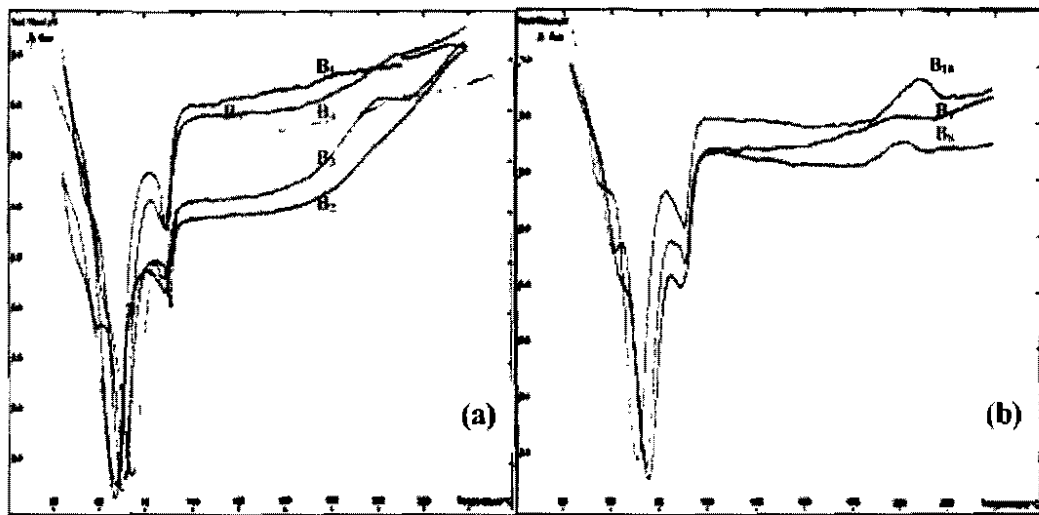

Fig. 2: DSC curves of binder systems (a) numbers 1, 2, 3, 4 & 5 and (b) numbers 8, 9 & 10.

B: binder.

Most of the heating curves for the formulated binder systems (nos. 1, 2, 4, 5, 9 & 10) show two endothermic peaks. It was found that, the addition of paraffin wax to the polymer component changes the melting temperature peaks of both. Thus the two endothermic temperature peaks represent the melting point of the wax (with or without SA) and the polymer in the binder system, respectively. Usually, the endothermic temperature peaks of SA and paraffin wax are overlapped and exhibit one melting temperature peak only [Lii, (1998)]. While, for the two binder systems nos. 3 & 8, three endothermic peaks were shown which represent the solid-solid transition (crystal phase separation) of PW_3 and the melting points of both PW_3 and EVA, respectively in the binder system (Fig. 2). This may be attributed to that PW_3 is not compatible with EVA at low temperatures.

The different melting temperatures of the binder systems ensure that when one binder component (wax) has melted, the remaining binder component (polymer) act as backbone support, retaining the shape of the molded part [Liu et al., (2001)].

Comparing the melting temperatures of the paraffin waxes (PW_1, PW_2, PW_3 & PW_4) and in their binder systems with those of the foreign one (PW_5) and in its binder systems, it can be observed that the melting point of the paraffin wax PW_3 is nearly the same of the foreign paraffin wax PW_5. Also melting points in their binder systems nos. 3 & 5, respectively are nearly similar. Moreover, it can be noticed that the melting points of the paraffin waxes PW_1 & PW_2 and consequently their melting points in their binder systems nos. 1 & 2 are lower than those of paraffin wax PW_5 and in its binder system no. 5. While the melting point of the paraffin wax PW_4 and in its binder systems nos. 4 & 9 are higher than the melting point of PW_5 and in its binder systems nos. 5 & 10 (Tables 3 & 4).

Also data in Table 3 indicate that, the melting temperatures of the paraffin waxes (PW_1, PW_2, PW_3, PW_4 & PW_5) obtained by DSC are accordance to those obtained by ASTM-938 (congealing points), whereas they are nearly the same or slightly higher than the congealing points of these paraffin waxes.

3.4. Thermo Gravimetric Analysis

The thermal stability of the binder components was characterized in terms of their temperatures of degradation. The TGA curves of the pure binder components are shown in Figure 3. It can be seen that, the degradation temperatures of the binder components range from 175-260, 206-313, 212-321, 206-320, 180-269 and 296-   500 ^{0}C for PW_1, PW_3, PW_4, PW_5, SA and EVA, respectively. Also, it can be noticed that PW_3 & PW_4 have nearly the same thermal stability as PW_5. PW_1 has the lowest one, while EVA has the highest thermal stability.

According to the thermal properties of the binder components, the mixing temperature was set higher than the highest melting point, but lower than the lowest degradation temperature of the binder components. Thus. from the previous data obtained by DSC and TGA analysis, it was found that the temperatures which can be used to establish the permissible mixing and molding of the formulated binder systems and feedstock are higher than the melting peak temperature of EVA (100 ^{0}C) and lower than the degradation temperature of PW_1 (175 ^{0}C). Consequently, the suitable mixing temperatures for formulating the. binder systems were chosen to be in the range (120- 140 ^{0}C).
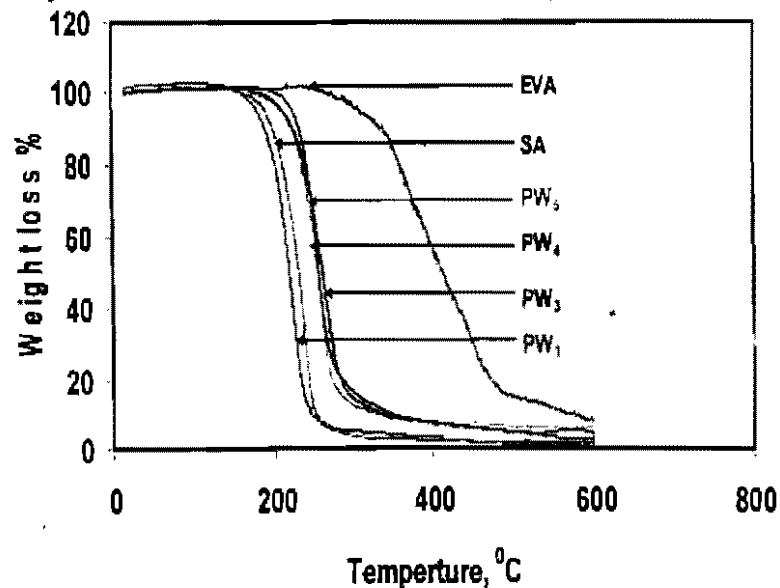

Fig. 3: TGA curves for pure binder components (PW_1. PW_3. PW_4. PW_5, SA and EVA)

3.5. Rheological Measurements

Generally, the binder is the main component controlling the mixing, molding and debinding operations. The essential requirement of a binder is to have low viscosity necessary for mixing. Thus, in order to choose the suitable formulated binder system for preparation a successful feedstock, the rheological properties of all the binder systems (1-10) were measured in terms of determining their viscosities in the temperatures ranges (120-140 ^{0}C) as a function of shear rate. Data are represented in Figures 4 (a, b & c).

It is known that when the viscosity of the binder is below 10 Pa.s, the feedstock including high powder volume does not show any processing problem during injection molding [Chung et al., (1989) and Park & Kim, (2001)]. However, when the viscosity becomes extremely low, jetting occurs during the injection molding process; thus the exact shape of the molded article is impossible [Song et al., (2005)]. Consequently, it can be found that the most suitable binder systems which confirm the previous requirements are the binder system no. 3 at temperature of 120 ^{0}C, the binder system no. 8 at temperature of 130 ^{0}C and the binder system no. 4 at temperature of 140 ^{0}C. The binder systems nos. 3 and 8 are rejected for molding process because the paraffin wax (PW$_3$) in both binders is not compatible with the polymer as discussed before. Also, the binder system no. 8 contains no SA; which is necessary to modify the powder wetting and creating a chemical bridge between the powder and the major binder constituents [German & Bose, (1997)]. Thus, it can be concluded that the binder system no. 4 (consisted of 35 wt. % EVA containing vinyl acetate content of 9 wt %, 62 wt % of PW$_4$ and 3 wt % of SA) is the preferable binder for powder injection molding. It has nearly the same viscosity as the well known binder system no. 5 (consisted of 35 wt. % EVA containing vinyl acetate content of 9.5 wt %, 62 wt. % of foreign paraffin wax PW$_5$ and 3 wt. % of SA).
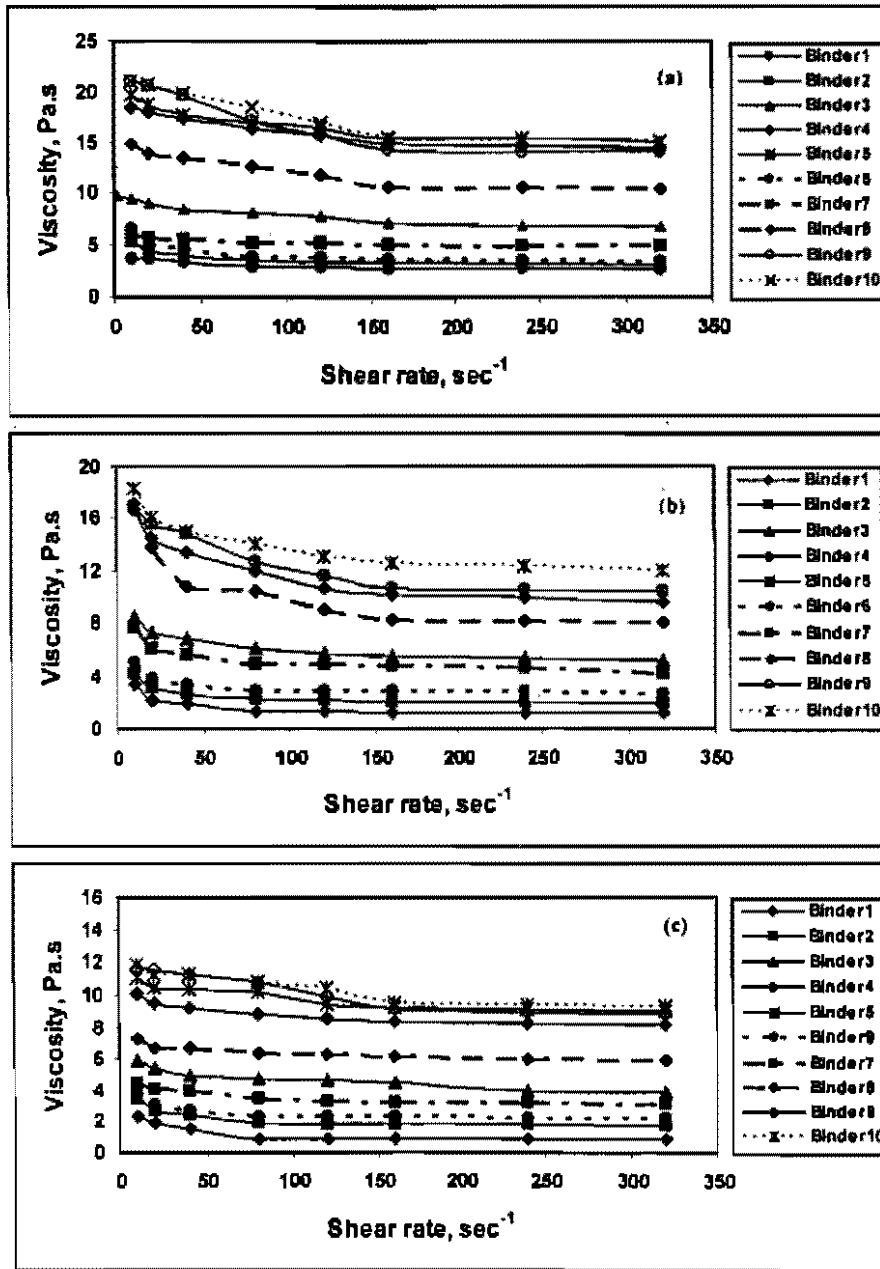
Fig. 4: Rheological characteristics of binder systems (a) at 120 ^{0}C, (b) at 130 ^{0}C and (c) at 140 ^{0}C.

CONCLUSION

From the previous study it can be concluded that, to select the suitable paraffin wax for formulation of proper wax-based binder, it is necessary to characterize it totally by physical, molecular and thermal tools of analysis. Also, the formulated binder systems should be characterized thermally and rheologically to select the best one. Data revealed that the formulated binder system no. 4 (consisted of 35 wt. % EVA containing vinyl acetate content of 9 wt %, 62 wt % of heavy paraffin wax PW_4 and 3 wt % of SA) provides an excellent wax-based binder system resemble the well known binder system no. 5 (consisted of 35 wt. % EVA containing vinyl acetate content of 9.5 wt %, 62 wt. % of foreign paraffin wax PW_5 and 3 wt. % of SA). This binder system has good compatibility, rheological properties as it has viscosity below 10 Pa.s which is suitable to fabricate homogenous feedstock during compression or injection molding process.

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تأثير خواص الشمع البارافينى على تكوين وريولوجية
المواد الرابطة المعتمدة على الشموع

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تم دراسة تأثير الشموع البارافينية على خواص و ريولوجية (Rheology) المـواد
الرابطة المعتمدة على الشموع (Wax-based binders) . و قد أختيرت أربع عينات مـن
الشموع البارافينية المحلية (1 ، 2 ، 3 ، 4) و قورنت بعينة الشمع البارافينى المستورد (5).

تم تكوين عشرة مواد رابطة معتمدة على الشموع و ذلك بخلـط المـادة الرابطـة
الرئيسية (Major binder) المحتوية على الشمع البارافينى (Paraffin wax) مضـاف أو
غير مضاف إليه حمض الأستياريك (Stearic acid) مع المادة الرابطة الثانوية (البـوليمر)
(Ethylene-co-vinyl acetate) . وقد قورنت المواد الرابطة المحتويـة علـى الشـموع
المحلية أرقام 1 – 4 و 6 – 10 مع المادة الرابطة رقم 5 المحتوية على الشـمع البـارافينى
المستورد.

تم دراسة الخواص الفيزيائية و الجزيئية لجميع الشموع البارافينية. و أيضـا تم تعيين
الخواص الحرارية لمكونات المواد الرابطة و المواد الرابطة المحضرة بأسـتخدام المسـعر
الماسح التباينى (DSC) و جهاز قياس الوزن الحـرارى (TGA). و أخيــرا تـم دراسـة
الخواص الريولوجية للمواد الرابطة المحضرة و ذلك بتعيين لزوجتها (Viscosity).

قد بينت النتائج أن المادة الرابطة المحضرة رقم 4 المكونة من 35 % من البوليمر
(Ethylene-co-vinyl acetate) (المحتوى على 9 % من أسيتات الفينيل) ، 62 % مـن
الشمع البارافينى المحلى رقم 4 (PW₄) و 3 % من حمض الأستياريك هى أفضـل مـادة
رابطة تصلح لعمل قولبة للبودرة بالحقن (Powder injection molding).